

SUPPORTING INFORMATION

Enantioselective organocatalytic syntheses of α -selenated α - and β -amino acid derivatives

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1. General Information

^1H -, ^{13}C - and ^{19}F -NMR spectra were recorded on a Bruker Avance III 300 MHz spectrometer with a broad band observe probe and a sample changer for 16 samples, a Bruker Avance DRX 500 MHz spectrometer and on a Bruker Avance III 700 MHz spectrometer with an Ascend magnet and TCI cryoprobe, which are property of the Austro-Czech NMR-Research Center “RERI-uasb”. NMR spectra were referenced on the solvent peak and chemical shifts are given in ppm.

High resolution mass spectra were obtained using an Agilent 6520 Q-TOF mass spectrometer with an ESI source. Analyses were made in the positive ionization mode if not otherwise stated. Purine (exact mass for $[M+H]^+$ = 121.050873) and 1,2,3,4,5,6-hexakis(2,2,3,3-tetrafluoropropoxy)-1,3,5,2,4,6-triazatriphosphinane (exact mass for $[M+H]^+$ = 922.009798) were used for internal mass calibration.

HPLC was performed using a Thermo Scientific Dionex Ultimate 3000 or a Shimadzu Prominence system with diode array detector with a CHIRALPAK AD-H, OD-H or YMC CHIRAL ART Cellulose-SB (250 \times 4.6 mm, 5 μm) chiral stationary phase. Optical rotations were recorded on a Schmidt + Haensch Polarimeter Model UniPol L1000 at 589 nm.

All chemicals were purchased from commercial suppliers and used without further purification unless otherwise stated. Dry solvents were obtained from an MBraun-SPS-800 solvent purification system. All reactions were carried out under argon atmosphere and in absence of light, unless stated otherwise.

Azlactones¹ **1**, α -Aryl-Isoxazolidin-5-ones² **2** and selenation reagents³ **5** were prepared following established procedures.

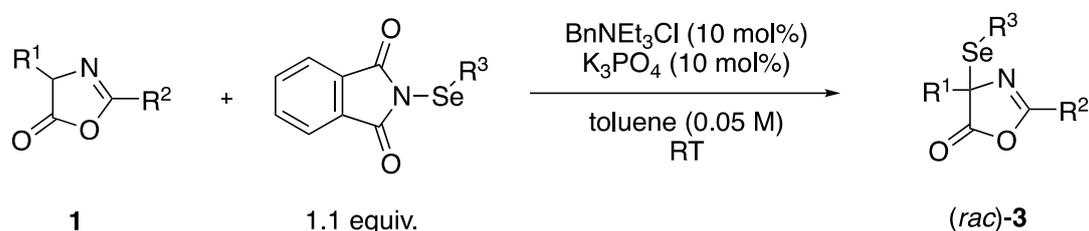
¹ (a) C. Macovai, P. Vicennati, J. Quinton, M.-C. Nevers, H. Volland, C. Créminon and F. Taran, *Chem. Commun.* 2012, **48**, 4411-4413; (b) A. D. Melhado, M. Luparia and F. D. Toste, *J. Am. Chem. Soc.* 2007, **129**, 42, 12638-12639; (c) D. N. Le, J. Riedel, N. Kozlyuk, R. W. Martin and V. M. Dong, *Org. Lett.* 2017, **19**, 1, 114-117

² M. N. Oliveira, S. Arseniyadis and J. Cossy, *Chem. Eur. J.* 2018, **24**, 4810-4814.

³ (a) X.-Y. Wang, Y.-F. Zhong, Z.-Y. Mo, S.-H. Wu, Y.-L. Xu, H.-T. Tang and Y.-M. Pan, *Adv. Synth. Catal.* 2021, **363**, 208-214; (b) T. Hori and K. B. Sharpless, *J. Org. Chem.* 1979, **44**, 4208-4210.

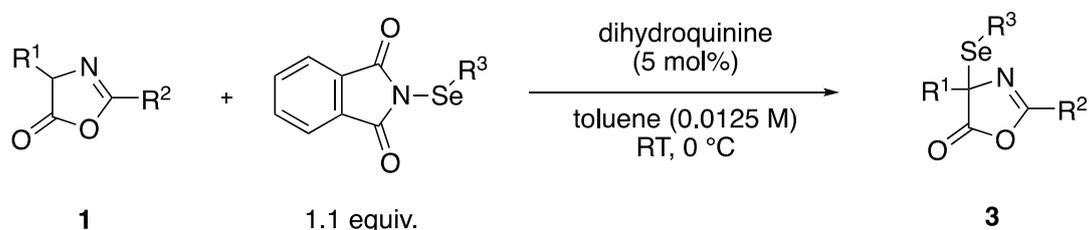
2. Synthesis of α -Selenated Azlactones 3

2.1 General Racemic Procedure



To a solution of azlactone (**1**, 1 equiv.), benzyltriethylammonium chloride (BTEAC, 10 mol%) and K_3PO_4 (10 mol%) in toluene (0.05 M with respect to azlactone substrate **1**) under an atmosphere of argon was added the corresponding *N*-(Aryl-seleno)phthalimide (1.1 equiv.) at room temperature and under exclusion of light. The reaction mixture was stirred for 1 h under these conditions and the reaction was monitored by thin layer chromatography using heptanes:EtOAc (3.5:1) as mobile phase. After filtration through a pad of Na_2SO_4 the crude product was subjected to silica gel column chromatography (eluent: heptanes:EtOAc) for purification.

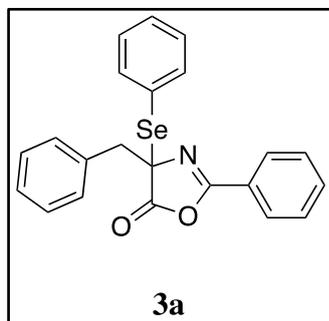
2.2 General Enantioselective Procedure



To a solution of azlactone (**1**, 1 equiv.) and dihydroquinine (5 mol%) in toluene (0.0125 M related to azlactone **1**) under an atmosphere of argon was added the corresponding *N*-(Aryl-seleno)phthalimide (1.1 equiv.) at 0 °C and under exclusion of light. The reaction mixture was stirred for 1 h under these conditions and the reaction was monitored by thin layer chromatography using heptanes:EtOAc (3.5:1) as mobile phase. After filtration through a pad of Na_2SO_4 the crude product was subjected to silica gel column chromatography (eluent: heptanes:EtOAc) for purification.

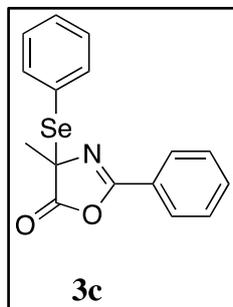
3. Characterization Data of α -Selenated Azlactones 3

4-Benzyl-2-phenyl-4-(phenylselanyl)oxazol-5(4H)-one (**3a**):

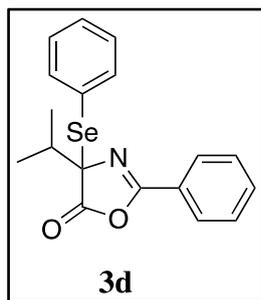


Compound **3a** was prepared according to the general procedure described in **2.2** and was obtained as a white solid (18.7 mg, 46 μ mol, 92% yield, e.r. = 89:11). **TLC** (heptanes:EtOAc = 3.5:1): R_f = 0.49 (UV). $[\alpha]_D^{24}$ = -65.1 (c 1.00, CHCl_3 , e.r. = 89:11). **$^1\text{H-NMR}$** (300 MHz, CDCl_3 , 298.0 K): δ / ppm = 7.57 (d, J = 9.0 Hz, 2H), 7.50 (d, J = 6.0 Hz, 2H), 7.39 (t, J = 9.0 Hz, 1H), 7.26 (t, J = 9.0 Hz, 2H), 7.19-7.14 (m, 4H), 7.12-7.02 (m, 4H), 3.54 (d, J = 12.0 Hz, 1H), 3.42 (d, J = 15.0 Hz, 1H). **$^{13}\text{C-NMR}$** (75 MHz, CDCl_3 , 298.0 K): δ / ppm = 176.4, 160.7, 138.3, 134.6, 132.8, 130.2, 130.2, 129.2, 128.7, 128.5, 127.9, 127.6, 125.3, 73.9, 41.3. **HRMS** (ESI-QTOF, MeOH) m/z : $[\text{M}+\text{H}]^+$ calculated for $\text{C}_{23}\text{H}_{17}\text{NO}_2\text{Se}$, 408.0498; found, 408.0500. **HPLC**: Chiralpak AD-H (n -hexane: i -PrOH = 20:1, flow rate 0.3 mL/min, 10 $^\circ\text{C}$, λ = 220 nm), retention times $t_{\text{R}}(\text{minor})$ = 26.4 min, $t_{\text{R}}(\text{major})$ = 27.8 min.

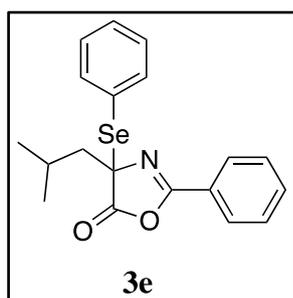
4-Methyl-2-phenyl-4-(phenylselanyl)oxazol-5(4H)-one (**3c**):



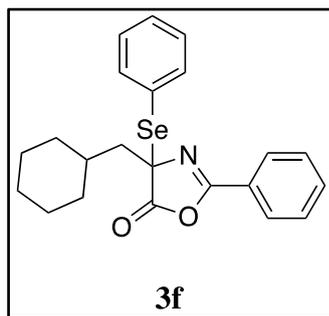
Compound **3c** was prepared according to the general procedure described in **2.2** and was obtained as a white solid (14.2 mg, 43 μ mol, 86% yield, e.r. = 94:6). **TLC** (heptanes:EtOAc = 3.5:1): R_f = 0.57 (UV). $[\alpha]_D^{24}$ = -30.1 (c 1.00, CHCl_3 , e.r. = 94:6). **$^1\text{H-NMR}$** (700 MHz, CDCl_3 , 298.0 K): δ / ppm = 7.76 (d, J = 7.0 Hz, 2H), 7.57 (d, J = 14.0 Hz, 2H), 7.53 (t, J = 14.0 Hz, 1H), 7.40 (t, J = 7.0 Hz, 2H), 7.25 (t, J = 7.0 Hz, 1H), 7.14 (t, J = 7.0 Hz, 2H), 1.93 (s, 3H). **$^{13}\text{C-NMR}$** (176 MHz, CDCl_3 , 298.0 K): δ / ppm = 177.7, 160.6, 138.1, 133.0, 130.2, 129.1, 128.8, 127.9, 125.8, 125.3, 69.2, 21.9. **HRMS** (ESI-QTOF, MeOH) m/z : $[\text{M}+\text{H}]^+$ calculated for $\text{C}_{16}\text{H}_{14}\text{NO}_2\text{Se}$, 332.0185; found, 332.0187. **HPLC**: Chiralpak AD-H (n -hexane: i -PrOH = 20:1, flow rate 0.3 mL/min, 10 $^\circ\text{C}$, λ = 220 nm), retention times $t_{\text{R}}(\text{major})$ = 24.8 min, $t_{\text{R}}(\text{minor})$ = 30.3 min.

4-Isopropyl- 2-phenyl-4-(phenylselenanyl)oxazol-5(4H)-one (3d):

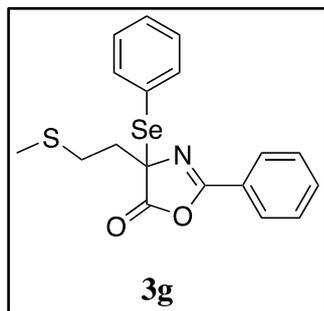
Compound **3d** was prepared according to the general procedure described in **2.2** and was obtained as a colourless solid (11.3 mg, 32 μ mol, 63% yield, e.r. = 79:21). **TLC** (heptanes:EtOAc = 3.5:1): R_f = 0.44 (UV). $[\alpha]_D^{23}$ = -11.0 (c 1.00, CHCl_3 , e.r. = 79:21). **$^1\text{H-NMR}$** (500 MHz, CDCl_3 , 298.0 K): δ / ppm = 7.75 (d, J = 7.9 Hz, 2H), 7.55-7.50 (m, 3H), 7.40 (t, J = 7.7 Hz, 2H), 7.23 (t, J = 7.3 Hz, 1H), 7.11 (t, J = 7.7 Hz, 2H), 2.54 (sep, J = 6.9 Hz, 1H) 1.33 (d, J = 6.7 Hz, 3H), 1.05 (d, J = 6.7 Hz, 3H). **$^{13}\text{C-NMR}$** (126 MHz, CDCl_3 , 298.0 K): δ / ppm = 176.9, 160.7, 138.4, 132.8, 130.0, 129.1, 128.7, 127.9, 125.5, 125.2, 79.2, 33.9, 18.6, 18.5. **HRMS** (ESI-QTOF, MeOH) m/z : $[\text{M}+\text{H}]^+$ calculated for $\text{C}_{18}\text{H}_{18}\text{NO}_2\text{Se}$, 360.0498; found, 360.0501. **HPLC**: Chiralpak AD-H (n -hexane: i -PrOH = 20:1, flow rate 0.3 mL/min, 10 $^\circ\text{C}$, λ = 220 nm), retention times t_R (major) = 17.1 min, t_R (minor) = 18.6 min.

4-Isobutyl- 2-phenyl-4-(phenylselenanyl)oxazol-5(4H)-one (3e):

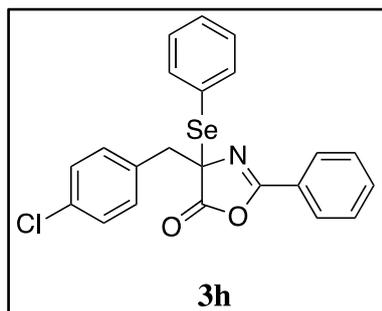
Compound **3e** was prepared according to the general procedure described in **2.2** and was obtained as a colourless solid (18.2 mg, 49 μ mol, 98% yield, e.r. = 92:8). **TLC** (heptanes:EtOAc = 3.5:1): R_f = 0.45 (UV). $[\alpha]_D^{23}$ = -93.3 (c 1.00, CHCl_3 , e.r. = 92:8). **$^1\text{H-NMR}$** (500 MHz, CDCl_3 , 298.0 K): δ / ppm = 7.74 (d, J = 7.2 Hz, 2H), 7.54-7.50 (m, 3H), 7.39 (t, J = 7.8 Hz, 2H), 7.24 (t, J = 7.4 Hz, 1H), 7.10 (t, J = 7.7 Hz, 2H), 2.32 (dd, J_1 = 5.6 Hz, J_2 = 14.2 Hz, 1H), 2.17 (dd, J_1 = 7.7 Hz, J_2 = 14.1 Hz, 1H), 1.83 (sep, J = 6.6 Hz, 1H), 0.95 (d, J = 6.6 Hz, 3H), 0.85 (d, J = 6.6 Hz, 3H). **$^{13}\text{C-NMR}$** (126 MHz, CDCl_3 , 298.0 K): δ / ppm = 177.5, 160.3, 138.3, 132.8, 130.2, 129.0, 128.7, 127.8, 125.4, 125.4, 73.4, 43.3, 27.0, 23.6, 22.7. **HRMS** (ESI-QTOF, MeOH) m/z : $[\text{M}+\text{H}]^+$ calculated for $\text{C}_{19}\text{H}_{20}\text{NO}_2\text{Se}$, 374.0654; found, 374.0657. **HPLC**: YMC Chiral Art Cellulose SB (n -hexane: i -PrOH = 80:1, flow rate 0.2 mL/min, 10 $^\circ\text{C}$, λ = 220 nm), retention times t_R (major) = 28.9 min, t_R (minor) = 30.0 min.

4-(Cyclohexylmethyl)-2-phenyl-4-(phenylselenanyl)oxazol-5(4H)-one (3f):

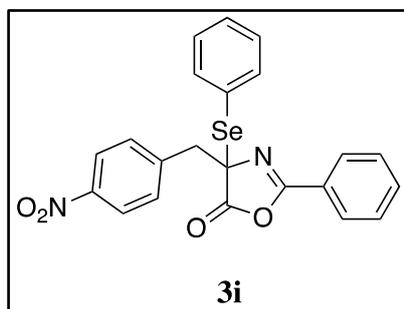
Compound **3f** was prepared according to the general procedure described in **2.2** and was obtained as a white solid (19.4 mg, 47 μmol , 94% yield, e.r. = 93:7). **TLC** (heptanes:EtOAc = 3.5:1): R_f = 0.63 (UV). $[\alpha]_D^{23}$ = -109.1 (c 1.00, CHCl_3 , e.r. = 93:7). **$^1\text{H-NMR}$** (300 MHz, CDCl_3 , 298.0 K): δ / ppm = 7.75-7.72 (m, 2H), 7.55-7.50 (m, 3H), 7.40 (t, J = 7.7 Hz, 2H), 7.23 (t, J = 7.4 Hz, 1H), 7.10 (t, J = 7.6 Hz, 2H), 2.33-2.15 (m, 2H), 1.73-1.47 (m, 6H), 1.19-0.88 (m, 5H). **$^{13}\text{C-NMR}$** (75 MHz, CDCl_3 , 298.0 K): δ / ppm = 177.6, 160.3, 138.3, 132.8, 130.2, 129.1, 128.8, 127.9, 125.5, 125.5, 77.4, 73.4, 42.2, 36.1, 34.1, 33.2, 26.2, 26.0. **HRMS** (ESI-QTOF, MeOH) m/z : $[\text{M}+\text{H}]^+$ calculated for $\text{C}_{22}\text{H}_{24}\text{NO}_2\text{Se}$, 414.0967; found, 414.0965. **HPLC**: Chiralpak AD-H (n -hexane: i -PrOH = 20:1, flow rate 0.7 mL/min, 10 $^\circ\text{C}$, λ = 220 nm), retention times $t_R(\text{minor})$ = 20.6 min, $t_R(\text{major})$ = 22.9 min.

4-(2-(Methylthio)ethyl)-2-phenyl-4-(phenylselenanyl)oxazol-5(4H)-one (3g):

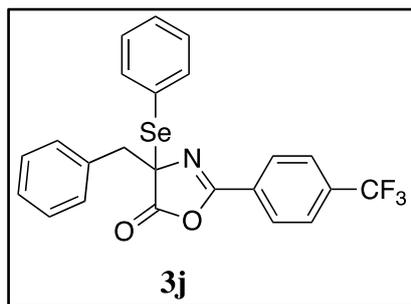
Compound **3g** was prepared according to the general procedure described in **2.2** and was obtained as a colourless solid (19.1 mg, 49 μmol , 98% yield, e.r. = 93:7). **TLC** (heptanes:EtOAc = 3.5:1): R_f = 0.43 (UV). $[\alpha]_D^{24}$ = -84.9 (c 1.00, CHCl_3 , e.r. = 93:7). **$^1\text{H-NMR}$** (300 MHz, CDCl_3 , 298.0 K): δ / ppm = 7.77-7.75 (m, 2H), 7.57-7.51 (m, 3H), 7.40 (t, J = 7.8 Hz, 2H), 7.29-7.23 (m, 1H), 7.14 (t, J = 7.6 Hz, 2H), 2.66-2.56 (m, 4H), 2.05 (s, 3H). **$^{13}\text{C-NMR}$** (75 MHz, CDCl_3 , 298.0 K): δ / ppm = 177.1, 161.5, 138.3, 133.0, 130.3, 129.2, 128.8, 128.0, 125.4, 125.0, 72.3, 34.1, 30.3, 15.1. **HRMS** (ESI-QTOF, MeOH) m/z : $[\text{M}+\text{H}]^+$ calculated for $\text{C}_{18}\text{H}_{18}\text{NO}_2\text{SSe}$, 392.0218; found, 392.0219. **HPLC**: Chiralpak AD-H (n -hexane: i -PrOH = 20:1, flow rate 0.3 mL/min, 10 $^\circ\text{C}$, λ = 220 nm), retention times $t_R(\text{minor})$ = 31.7 min, $t_R(\text{major})$ = 33.2 min.

4-(4-Chlorobenzyl)- 2-phenyl-4-(phenylselanyl)oxazol-5(4H)-one (3h):

Compound **3h** was prepared according to the general procedure described in **2.2** and was obtained as a colourless solid (21.8 mg, 50 μ mol, 98% yield, e.r. = 82:18). **TLC** (heptanes:EtOAc = 3.5:1): R_f = 0.49 (UV). $[\alpha]_D^{24}$ = -2.3 (c 1.00, CHCl_3 , e.r. = 82:18). **$^1\text{H-NMR}$** (300 MHz, CDCl_3 , 298.0 K): δ / ppm = 7.62 (d, J = 6.0 Hz, 2H), 7.54 (d, J = 9.0 Hz, 2H), 7.48-7.43 (m, 1H), 7.32 (t, J = 9.0 Hz, 2H), 7.24-7.19 (m, 1H), 7.17-7.08 (m, 6H), 3.55 (d, J = 12.0 Hz, 1H), 3.42 (d, J = 12.0 Hz, 1H). **$^{13}\text{C-NMR}$** (75 MHz, CDCl_3 , 298.0 K): δ / ppm = 176.3, 160.9, 138.3, 134.5, 133.6, 133.0, 133.0, 131.6, 130.3, 129.2, 128.7, 127.9, 125.2, 125.1, 123.7, 73.4, 40.5. **HRMS** (ESI-QTOF, MeOH) m/z : $[\text{M}+\text{H}]^+$ calculated for $\text{C}_{22}\text{H}_{17}\text{ClNO}_2\text{Se}$, 442.0108; found, 442.0106. **HPLC**: Chiralpak AD-H (n -hexane: i -PrOH = 20:1, flow rate 0.3 mL/min, 10 $^\circ\text{C}$, λ = 220 nm), retention times $t_R(\text{major})$ = 31.4 min, $t_R(\text{minor})$ = 33.9 min.

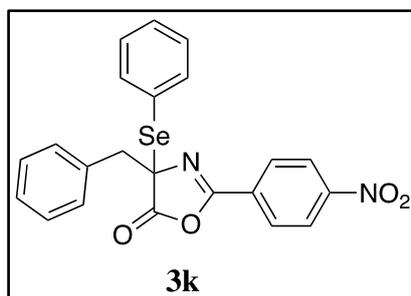
4-(4-Nitrobenzyl)- 2-phenyl-4-(phenylselanyl)oxazol-5(4H)-one (3i):

Compound **3i** was prepared according to the general procedure described in **2.2** and was obtained as a light-yellow solid (21.0 mg, 47 μ mol, 93% yield, e.r. = 69:31). **TLC** (heptanes:EtOAc = 3.5:1): R_f = 0.42 (UV). $[\alpha]_D^{24}$ = -27.2 (c 1.00, CHCl_3 , e.r. = 69:31). **$^1\text{H-NMR}$** (300 MHz, CDCl_3 , 298.0 K): δ / ppm = 8.08 (d, J = 8.8 Hz, 2H), 7.68-7.65 (m, 2H), 7.59-7.56 (m, 2H), 7.54-7.49 (m, 1H), 7.44 (d, J = 8.8 Hz, 2H), 7.37 (t, J = 7.8 Hz, 2H), 7.31-7.25 (m, 1H), 7.16 (t, J = 7.5 Hz, 2H), 3.72 (d, J = 13.7 Hz, 1H), 3.58 (d, J = 13.7 Hz, 1H). **$^{13}\text{C-NMR}$** (75 MHz, CDCl_3 , 298.0 K): δ / ppm = 176.1, 161.2, 147.5, 142.0, 138.3, 133.3, 131.2, 130.5, 129.3, 128.8, 127.9, 124.9, 124.8, 123.7, 72.6, 40.8. **HRMS** (ESI-QTOF, MeOH) m/z : $[\text{M}+\text{H}]^+$ calculated for $\text{C}_{22}\text{H}_{17}\text{N}_2\text{O}_4\text{Se}$, 453.0348; found, 453.0352. **HPLC**: Chiralpak AD-H (n -hexane: i -PrOH = 20:1, flow rate 0.7 mL/min, 10 $^\circ\text{C}$, λ = 220 nm), retention times $t_R(\text{minor})$ = 30.0 min, $t_R(\text{major})$ = 32.4 min.

4-Benzyl-4-(phenylselenanyl)-2-(4-(trifluoromethyl)phenyl)oxazol-5(4H)-one (3j):

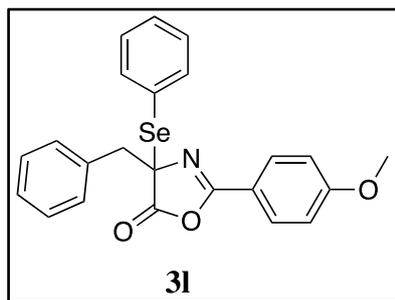
Compound **3j** was prepared according to the general procedure described in **2.2** and was obtained as a colourless solid (19.2 mg, 41 μ mol, 81% yield, e.r. = 72:28). **TLC** (heptanes:EtOAc = 3.5:1): R_f = 0.45 (UV). $[\alpha]_D^{24}$ = -46.4 (c 1.00, CHCl₃, e.r. = 72:28). **¹H-NMR** (700 MHz, CDCl₃, 298.0 K): δ / ppm = 7.76 (d, J = 14.0 Hz, 2H), 7.62 (d, J = 7.0 Hz, 2H), 7.58 (d, J = 7.0 Hz, 2H), 7.28-7.24 (m, 3H), 7.21 (t, J = 7.0 Hz, 2H), 7.19-7.14 (m, 3H), 3.63 (d, J = 14.0 Hz, 1H), 3.54 (d, J = 7.0 Hz, 1H). **¹³C-NMR** (176 MHz, CDCl₃, 298.0 K): δ / ppm = 175.9, 159.4, 138.3, 134.3, 130.4, 130.2, 129.3, 128.6, 128.2, 127.7, 125.7, 125.7, 125.1, 122.8, 73.8, 41.1. **¹⁹F-NMR** (471 MHz, CDCl₃, 298.0 K): δ / ppm = -63.21.

HRMS (ESI-QTOF, MeOH) m/z : $[M+H]^+$ calculated for C₂₃H₁₇F₃NO₂Se, 476.0371; found, 476.0377. **HPLC**: Chiralpak AD-H (*n*-hexane:*i*-PrOH = 20:1, flow rate 0.3 mL/min, 10 °C, λ = 220 nm), retention times $t_{R(\text{minor})}$ = 21.3 min, $t_{R(\text{major})}$ = 26.3 min.

4-Benzyl-2-(4-nitrophenyl)-4-(phenylselenanyl)oxazol-5(4H)-one (3k):

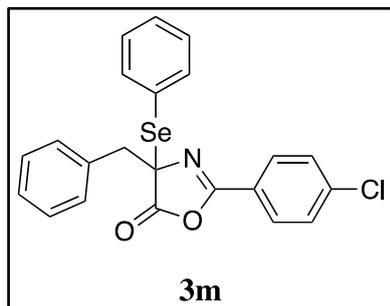
Compound **3k** was prepared according to the general procedure described in **2.2** and was obtained as a yellow solid (12.2 mg, 27 μ mol, 54% yield, e.r. = 63:37). **TLC** (heptanes:EtOAc = 3.5:1): R_f = 0.52 (UV). $[\alpha]_D^{23}$ = -23.1 (c 1.00, CHCl₃, e.r. = 63:37). **¹H-NMR** (300 MHz, CDCl₃, 298.0 K): δ / ppm = 8.20 (d, J = 9.1 Hz, 2H), 7.81 (d, J = 8.7 Hz, 2H), 7.58-7.55 (m, 2H), 7.29-7.12 (m, 8H), 3.65 (d, J = 13.7 Hz, 1H), 3.54 (d, J = 13.7 Hz, 1H). **¹³C-NMR** (75 MHz, CDCl₃, 298.0 K): δ / ppm = 175.6, 158.8, 150.3, 138.3, 134.3, 130.8, 130.5, 130.2, 129.3, 128.8, 128.6, 127.8, 125.1, 123.9, 73.8, 41.0. **HRMS** (ESI-QTOF, MeOH) m/z : $[M+H]^+$ calculated for C₂₂H₁₇N₂O₄Se, 453.0348; found, 453.0343.

HPLC: Chiralpak AD-H (*n*-hexane:*i*-PrOH = 20:1, flow rate 0.7 mL/min, 10 °C, λ = 220 nm), retention times $t_{R(\text{minor})}$ = 22.5 min, $t_{R(\text{major})}$ = 29.0 min.

4-Benzyl-2-(4-methoxyphenyl)-4-(phenylselenanyl)oxazol-5(4H)-one (3l):

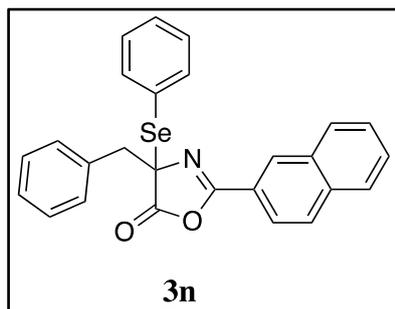
Compound **3l** was prepared according to the general procedure described in **2.2** and was obtained as a colourless solid (18.8 mg, 43 μ mol, 86% yield, e.r. = 91:9). **TLC** (heptanes:EtOAc = 3.5:1): R_f = 0.20 (UV). $[\alpha]_D^{24}$ = -44.9 (c 1.00, CHCl_3 , e.r. = 91:9). **$^1\text{H-NMR}$** (300 MHz, CDCl_3 , 298.0 K): δ / ppm = 7.63-7.58 (m, 4H), 7.29-7.12 (m, 8H),

6.84 (d, J = 9.0 Hz, 2H), 3.83 (s, 3H), 3.62 (d, J = 13.6 Hz, 1H), 3.48 (d, J = 13.6 Hz, 1H). **$^{13}\text{C-NMR}$** (75 MHz, CDCl_3 , 298.0 K): δ / ppm = 176.6, 163.3, 160.5, 138.2, 134.7, 130.3, 130.1, 129.8, 129.1, 128.5, 127.5, 125.5, 117.6, 114.1, 74.0, 55.6, 41.4. **HRMS** (ESI-QTOF, MeOH) m/z : $[\text{M}+\text{H}]^+$ calculated for $\text{C}_{23}\text{H}_{20}\text{NO}_3\text{Se}$, 438.0603; found, 438.0605. **HPLC**: Chiralpak AD-H (n -hexane: i -PrOH = 20:1, flow rate 0.7 mL/min, 10 $^\circ\text{C}$, λ = 270 nm), retention times $t_{\text{R}}(\text{minor})$ = 18.2 min, $t_{\text{R}}(\text{major})$ = 23.3 min.

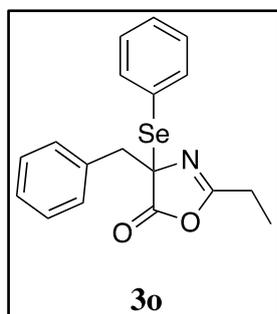
4-Benzyl-2-(4-chlorophenyl)-4-(phenylselenanyl)oxazol-5(4H)-one (3m):

Compound **3m** was prepared according to the general procedure described in **2.2** and was obtained as a colourless solid (17.0 mg, 39 μ mol, 77% yield, e.r. = 74:26). **TLC** (heptanes:EtOAc = 3.5:1): R_f = 0.58 (UV). $[\alpha]_D^{24}$ = -37.7 (c 1.00, CHCl_3 , e.r. = 74:26). **$^1\text{H-NMR}$** (300 MHz, CDCl_3 , 298.0 K): δ / ppm = 7.60-7.55 (m, 4H), 7.35-7.31 (m, 2H),

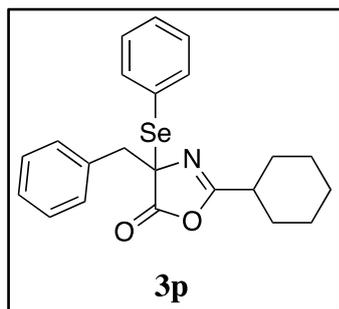
7.27-7.12 (m, 8H), 3.62 (d, J = 13.7 Hz, 1H), 3.50 (d, J = 13.7 Hz, 1H). **$^{13}\text{C-NMR}$** (75 MHz, CDCl_3 , 298.0 K): δ / ppm = 176.1, 159.8, 139.3, 138.3, 134.5, 130.3, 130.2, 129.2, 129.2, 128.6, 127.7, 125.2, 123.7, 73.9, 41.2. **HRMS** (ESI-QTOF, MeOH) m/z : $[\text{M}+\text{H}]^+$ calculated for $\text{C}_{22}\text{H}_{17}\text{ClNO}_2\text{Se}$, 442.0108; found, 442.0108. **HPLC**: Chiralpak AD-H (n -hexane: i -PrOH = 20:1, flow rate 0.3 mL/min, 10 $^\circ\text{C}$, λ = 220 nm), retention times $t_{\text{R}}(\text{minor})$ = 25.1 min, $t_{\text{R}}(\text{major})$ = 33.2 min.

4-Benzyl-2-(naphthalen-2-yl)-4-(phenylselanyl)oxazol-5(4H)-one (3n):

Compound **3n** was prepared according to the general procedure described in **2.2** and was obtained as a light-yellow solid (18.7 mg, 41 μ mol, 82% yield, e.r. = 87:13). **TLC** (heptanes:EtOAc = 3.5:1): R_f = 0.47 (UV). $[\alpha]_D^{24}$ = -88.0 (c 1.00, CHCl_3 , e.r. = 87:13). **$^1\text{H-NMR}$** (300 MHz, CDCl_3 , 298.0 K): δ / ppm = 8.07 (s, 1H), 7.83 (t, J = 6.8 Hz, 4H), 7.64-7.61 (m, 2H), 7.58-7.49 (m, 2H), 7.31-7.28 (m, 2H), 7.24-7.09 (m, 6H), 3.68 (d, J = 13.5 Hz, 1H), 3.55 (d, J = 13.7 Hz, 1H). **$^{13}\text{C-NMR}$** (75 MHz, CDCl_3 , 298.0 K): δ / ppm = 176.4, 160.8, 138.2, 135.4, 134.6, 132.5, 130.2, 130.2, 129.4, 129.2, 129.2, 128.6, 128.5, 128.0, 127.6, 127.1, 125.3, 123.4, 122.5, 74.1, 41.3. **HRMS** (ESI-QTOF, MeOH) m/z : $[\text{M}+\text{H}]^+$ calculated for $\text{C}_{26}\text{H}_{20}\text{NO}_2\text{Se}$, 458.0654; found, 458.0649. **HPLC**: Chiralpak AD-H (n -hexane: i -PrOH = 20:1, flow rate 0.7 mL/min, 10 $^\circ\text{C}$, λ = 220 nm), retention times $t_R(\text{minor})$ = 13.7 min, $t_R(\text{major})$ = 16.2 min.

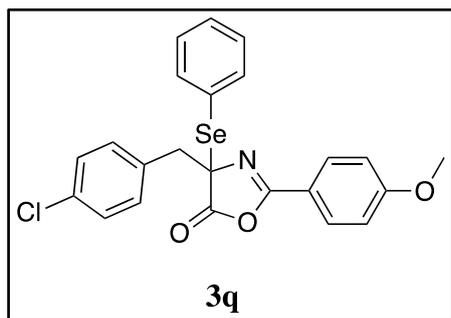
4-Benzyl-2-ethyl-4-(phenylselanyl)oxazol-5(4H)-one (3o):

Compound **3o** was prepared according to the general procedure described in **2.2** and was obtained as a colourless solid (14.2 mg, 40 μ mol, 79% yield, e.r. = 53:47). **TLC** (heptanes:EtOAc = 3.5:1): R_f = 0.36 (UV). **$^1\text{H-NMR}$** (500 MHz, CDCl_3 , 298.0 K): δ / ppm = 7.63-7.60 (m, 2H), 7.42 (t, J = 7.7 Hz, 1H), 7.32 (t, J = 7.7 Hz, 2H), 7.25-7.19 (m, 5H), 3.48 (d, J = 13.5 Hz, 1H), 3.42 (d, J = 13.5 Hz, 1H), 2.02-1.88 (m, 2H), 0.84 (t, J = 7.7 Hz, 3H). **$^{13}\text{C-NMR}$** (126 MHz, CDCl_3 , 298.0 K): δ / ppm = 177.0, 166.0, 138.3, 134.4, 130.3, 130.2, 129.3, 128.5, 127.6, 125.3, 72.7, 41.0, 22.1, 8.8. **HPLC**: Chiralpak AD-H (n -hexane: i -PrOH = 20:1, flow rate 0.3 mL/min, 10 $^\circ\text{C}$, λ = 220 nm), retention times $t_R(\text{major})$ = 21.5 min, $t_R(\text{minor})$ = 22.6 min.

4-Benzyl-2-cyclohexyl-4-(phenylselenanyl)oxazol-5(4H)-one (3p):

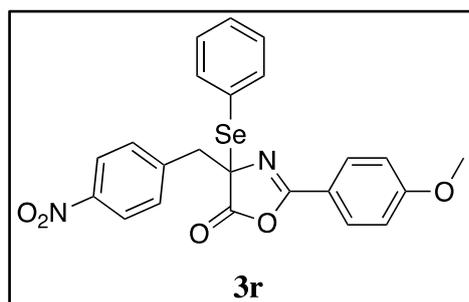
Compound **3p** was prepared according to the general procedure described in **2.2** and was obtained as a colourless solid (6.8 mg, 17 μ mol, 33% yield, e.r. = 53:47). **TLC** (heptanes:EtOAc = 3.5:1): R_f = 0.57 (UV). **$^1\text{H-NMR}$** (500 MHz, CDCl_3 , 298.0 K): δ / ppm = 7.64-7.62 (m, 2H), 7.42-7.39 (m, 1H), 7.32 (t, J = 7.7 Hz, 2H), 7.25-7.17 (m, 5H), 3.47 (d, J = 13.6 Hz, 1H), 3.42 (d, J = 13.2 Hz,

1H), 1.93-1.89 (m, 1H), 1.59-1.51 (m, 4H), 1.42-1.36 (m, 3H), 1.10-0.98 (m, 3H). **HRMS** (ESI-QTOF, MeOH) m/z : $[\text{M}+\text{H}]^+$ calculated for $\text{C}_{22}\text{H}_{24}\text{NO}_2\text{Se}$, 414.0967; found, 414.0963. **HPLC**: Chiralpak AD-H (n -hexane: i -PrOH = 20:1, flow rate 0.3 mL/min, 10 $^\circ\text{C}$, λ = 220 nm), retention times $t_{\text{R}}(\text{major})$ = 19.2 min, $t_{\text{R}}(\text{minor})$ = 19.9 min.

4-(4-Chlorobenzyl)-2-(4-methoxyphenyl)-4-(phenylselenanyl)oxazol-5(4H)-one (3q):

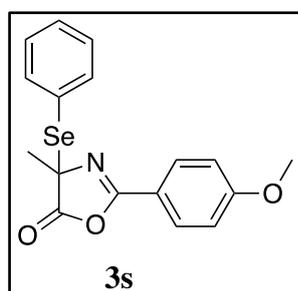
Compound **3q** was prepared according to the general procedure described in **2.2** and was obtained as a colourless solid (23.1 mg, 49 μ mol, 98% yield, e.r. = 87:13). **TLC** (heptanes:EtOAc = 3.5:1): R_f = 0.34 (UV). $[\alpha]_{\text{D}}^{23}$ = -7.5 (c 1.00, CHCl_3 , e.r. = 87:13). **$^1\text{H-NMR}$** (300 MHz, CDCl_3 , 298.0 K): δ / ppm = 7.72-7.65 (m, 4H),

7.38-7.33 (m, 2H), 7.29-7.21 (m, 5H), 6.94 (d, J = 8.9 Hz, 2H), 3.92 (s, 3H), 3.66 (d, J = 13.8 Hz, 1H), 3.52 (d, J = 13.8 Hz, 1H). **$^{13}\text{C-NMR}$** (75 MHz, CDCl_3 , 298.0 K): δ / ppm = 176.5, 163.5, 160.7, 138.3, 133.5, 133.2, 131.6, 130.2, 129.9, 129.2, 128.7, 125.3, 117.4, 114.2, 73.5, 55.6, 40.7. **HRMS** (ESI-QTOF, MeOH) m/z : $[\text{M}+\text{H}]^+$ calculated for $\text{C}_{23}\text{H}_{19}\text{ClNO}_3\text{Se}$, 472.0213; found, 472.0205. **HPLC**: Chiralpak AD-H (n -hexane: i -PrOH = 20:1, flow rate 0.7 mL/min, 10 $^\circ\text{C}$, λ = 220 nm), retention times $t_{\text{R}}(\text{minor})$ = 21.5 min, $t_{\text{R}}(\text{major})$ = 25.2 min.

2-(4-Methoxyphenyl)-4-(4-nitrobenzyl)-4-(phenylselenanyl)oxazol-5(4H)-one (3r):

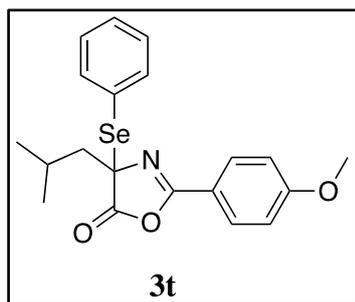
Compound **3r** was prepared according to the general procedure described in **2.2** and was obtained as a yellow solid (23.6 mg, 49 μ mol, 98% yield, e.r. = 79:21). **TLC** (heptanes:EtOAc = 3.5:1): R_f = 0.24 (UV). $[\alpha]_D^{23}$ = -21.5 (c 1.00, CHCl_3 , e.r. = 79:21). **$^1\text{H-NMR}$** (300 MHz, CDCl_3 , 298.0 K): δ / ppm = 8.07 (d, J = 8.7 Hz, 2H),

7.63-7.57 (m, 4H), 7.43 (d, J = 8.4 Hz, 2H), 7.31-7.24 (m, 1H), 7.17 (t, J = 7.7 Hz, 2H), 6.85 (d, J = 9.1 Hz, 2H), 3.84 (s, 3H), 3.70 (d, J = 13.3 Hz, 1H), 3.56 (d, J = 13.9 Hz, 1H). **$^{13}\text{C-NMR}$** (75 MHz, CDCl_3 , 298.0 K): δ / ppm = 176.2, 163.7, 161.0, 147.5, 142.2, 138.3, 131.2, 130.4, 129.9, 129.2, 125.1, 123.7, 117.0, 114.3, 72.8, 55.6, 41.0. **HRMS** (ESI-QTOF, MeOH) m/z : $[\text{M}+\text{H}]^+$ calculated for $\text{C}_{23}\text{H}_{19}\text{N}_2\text{O}_5\text{Se}$, 483.0454; found, 483.0462. **HPLC**: Chiralpak AD-H (n -hexane: i -PrOH = 20:1, flow rate 1.2 mL/min, 10 $^\circ\text{C}$, λ = 220 nm), retention times $t_{\text{R}}(\text{minor})$ = 28.0 min, $t_{\text{R}}(\text{major})$ = 32.2 min.

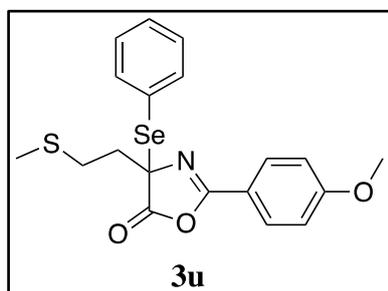
2-(4-Methoxyphenyl)-4-methyl-4-(phenylselenanyl)oxazol-5(4H)-one (3s):

Compound **3s** was prepared according to the general procedure described in **2.2** and was obtained as a colourless solid (5.9 mg, 17 μ mol, 33% yield, e.r. = 71:29). **TLC** (heptanes:EtOAc = 3.5:1): R_f = 0.28 (UV). $[\alpha]_D^{23}$ = +18.4 (c 0.47, CHCl_3 , e.r. = 71:29). **$^1\text{H-NMR}$** (300 MHz, CDCl_3 , 298.0 K): δ / ppm = 7.71 (d, J = 8.5 Hz, 2H), 7.57 (d, J = 7.7 Hz, 2H), 7.28-7.24 (m, 1H), 7.15 (t, J = 7.4 Hz, 2H),

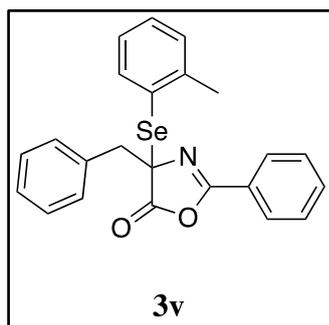
6.89 (d, J = 8.5 Hz, 2H), 3.86 (s, 3H), 1.92 (s, 3H). **HRMS** (ESI-QTOF, MeOH) m/z : $[\text{M}+\text{H}]^+$ calculated for $\text{C}_{17}\text{H}_{16}\text{NO}_3\text{Se}$, 362.0290; found, 362.0294. **HPLC**: Chiralpak AD-H (n -hexane: i -PrOH = 20:1, flow rate 0.7 mL/min, 10 $^\circ\text{C}$, λ = 220 nm), retention times $t_{\text{R}}(\text{minor})$ = 16.7 min, $t_{\text{R}}(\text{major})$ = 17.8 min.

4-Isobutyl-2-(4-methoxyphenyl)-4-(phenylselenanyl)oxazol-5(4H)-one (3t):

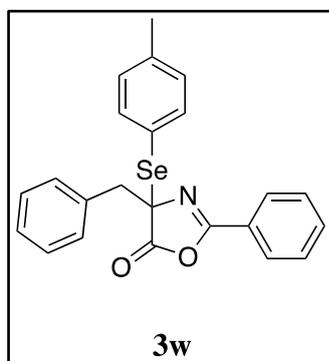
Compound **3t** was prepared according to the general procedure described in **2.2** and was obtained as a colourless solid (9.7 mg, 24 μ mol, 48% yield, e.r. = 59:41). **TLC** (heptanes:EtOAc = 3.5:1): R_f = 0.43 (UV). $[\alpha]_D^{23}$ = -32.6 (c 0.61, CHCl_3 , e.r. = 59:41). **$^1\text{H-NMR}$** (300 MHz, CDCl_3 , 298.0 K): δ / ppm = 7.70 (d, J = 8.7 Hz, 2H), 7.54 (d, J = 7.9 Hz, 2H), 7.26-7.23 (m, 1H), 7.12 (t, J = 7.7 Hz, 2H), 6.89 (d, J = 8.7 Hz, 2H), 3.86 (s, 3H), 2.33-2.29 (m, 1H), 2.18-2.14 (m, 1H), 1.82 (sep, J = 6.8 Hz, 1H), 0.94 (d, J = 6.8 Hz, 1H), 0.84 (d, J = 6.6 Hz, 1H). **$^{13}\text{C-NMR}$** (126 MHz, CDCl_3 , 298.0 K): δ / ppm = 177.7, 163.4, 160.2, 138.3, 130.1, 129.8, 129.0, 125.5, 117.8, 114.2, 73.6, 55.6, 43.5, 27.0, 23.7, 22.7. **HRMS** (ESI-QTOF, MeOH) m/z : $[\text{M}+\text{H}]^+$ calculated for $\text{C}_{20}\text{H}_{22}\text{NO}_3\text{Se}$, 404.0760; found, 404.0770. **HPLC**: Chiralpak AD-H (n -hexane: i -PrOH = 20:1, flow rate 0.7 mL/min, 10 $^\circ\text{C}$, λ = 220 nm), retention times $t_{\text{R}}(\text{minor})$ = 21.1 min, $t_{\text{R}}(\text{major})$ = 26.1 min.

2-(4-Methoxyphenyl)-4-(2-(methylthio)ethyl)-4-(phenylselenanyl)oxazol-5(4H)-one (3u):

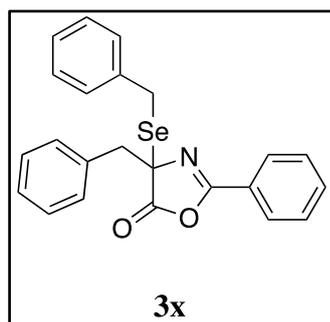
Compound **3u** was prepared according to the general procedure described in **2.2** and was obtained as a colourless solid (16.8 mg, 40 μ mol, 80% yield, e.r. = 91:9). **TLC** (heptanes:EtOAc = 3.5:1): R_f = 0.25 (UV). $[\alpha]_D^{23}$ = -32.1 (c 1.00, CHCl_3 , e.r. = 91:9). **$^1\text{H-NMR}$** (300 MHz, CDCl_3 , 298.0 K): δ / ppm = 7.72 (d, J = 8.6 Hz, 2H), 7.56 (d, J = 7.3 Hz, 2H), 7.29-7.24 (m, 1H), 7.15 (t, J = 8.0 Hz, 2H), 6.89 (d, J = 9.0 Hz, 2H), 3.86 (s, 3H), 2.71-2.52 (m, 4H), 2.04 (s, 3H). **$^{13}\text{C-NMR}$** (75 MHz, CDCl_3 , 298.0 K): δ / ppm = 177.2, 163.5, 161.2, 138.3, 130.2, 130.0, 129.1, 125.1, 117.7, 114.2, 72.5, 55.6, 34.3, 30.3, 15.1. **HRMS** (ESI-QTOF, MeOH) m/z : $[\text{M}+\text{H}]^+$ calculated for $\text{C}_{19}\text{H}_{20}\text{NO}_3\text{SSe}$, 422.0324; found, 422.0324. **HPLC**: Chiralpak AD-H (n -hexane: i -PrOH = 20:1, flow rate 0.7 mL/min, 10 $^\circ\text{C}$, λ = 220 nm), retention times $t_{\text{R}}(\text{minor})$ = 24.2 min, $t_{\text{R}}(\text{major})$ = 29.7 min.

4-Benzyl-2-phenyl-4-(*o*-tolylselenanyl)oxazol-5(4*H*)-one (3v):

Compound **3v** was prepared according to the general procedure described in **2.2** using *N*-(*o*-tolylseleno)phthalimide as a selenation agent and was obtained as a colourless solid (16.0 mg, 38 μ mol, 76% yield, e.r. = 88:12). **TLC** (heptanes:EtOAc = 3.5:1): R_f = 0.53 (UV). $[\alpha]_D^{24}$ = -84.4 (c 1.00, CHCl_3 , e.r. = 88:12). **$^1\text{H-NMR}$** (300 MHz, CDCl_3 , 298.0 K): δ / ppm = 7.65-7.62 (m, 2H), 7.58 (d, J = 7.5 Hz, 1H), 7.50-7.45 (m, 1H), 7.34 (t, J = 7.7 Hz, 2H), 7.29-7.16 (m, 5H), 7.12-7.10 (m, 2H), 6.92-6.87 (m, 1H), 3.66 (d, J = 13.8 Hz, 1H), 3.54 (d, J = 13.8 Hz, 1H), 2.52 (s, 3H). **$^{13}\text{C-NMR}$** (75 MHz, CDCl_3 , 298.0 K): δ / ppm = 176.7, 160.3, 144.4, 139.9, 134.7, 132.7, 130.7, 130.3, 130.2, 128.6, 128.5, 127.8, 127.5, 126.5, 126.5, 125.3, 74.2, 41.2, 23.7. **HRMS** (ESI-QTOF, MeOH) m/z : $[\text{M}+\text{H}]^+$ calculated for $\text{C}_{23}\text{H}_{20}\text{NO}_2\text{Se}$, 422.0654; found, 422.0650. **HPLC**: YMC Chiral Art Cellulose SB (*n*-hexane:*i*-PrOH = 20:1, flow rate 0.3 mL/min, 10 $^\circ\text{C}$, λ = 270 nm), retention times $t_{\text{R}}(\text{minor})$ = 24.1 min, $t_{\text{R}}(\text{major})$ = 25.3 min.

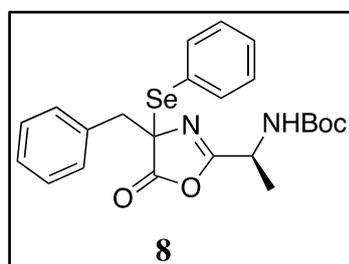
4-Benzyl-2-phenyl-4-(*p*-tolylselenanyl)oxazol-5(4*H*)-one (3w):

Compound **3w** was prepared according to the general procedure described in **2.2** using *N*-(*p*-tolylseleno)phthalimide as a selenation agent and was obtained as a colourless solid (10.1 mg, 24 μ mol, 48% yield, e.r. = 76:24). **TLC** (heptanes:EtOAc = 3.5:1): R_f = 0.44 (UV). $[\alpha]_D^{23}$ = -51.8 (c 0.80, CHCl_3 , e.r. = 76:24). **$^1\text{H-NMR}$** (300 MHz, CDCl_3 , 298.0 K): δ / ppm = 7.66 (d, J = 7.2 Hz, 2H), 7.51-7.44 (m, 3H), 7.36 (t, J = 7.8 Hz, 2H), 7.27-7.16 (m, 5H), 6.94 (d, J = 7.9 Hz, 2H), 3.62 (d, J = 13.8 Hz, 1H), 3.49 (d, J = 13.8 Hz, 1H), 2.20 (s, 3H). **$^{13}\text{C-NMR}$** (75 MHz, CDCl_3 , 298.0 K): δ / ppm = 176.5, 160.6, 140.6, 138.2, 134.7, 132.8, 130.2, 130.0, 128.6, 128.5, 127.9, 127.5, 125.4, 122.0, 73.7, 41.2, 21.3. **HRMS** (ESI-QTOF, MeOH) m/z : $[\text{M}+\text{H}]^+$ calculated for $\text{C}_{23}\text{H}_{20}\text{NO}_2\text{Se}$, 422.0654; found, 422.0658. **HPLC**: Chiralpak AD-H (*n*-hexane:*i*-PrOH = 20:1, flow rate 0.3 mL/min, 10 $^\circ\text{C}$, λ = 220 nm), retention times $t_{\text{R}}(\text{major})$ = 30.9 min, $t_{\text{R}}(\text{minor})$ = 32.8 min.

4-Benzyl-4-(benzylselanyl)-2-phenyloxazol-5(4H)-one (3x):

Compound **3x** was prepared according to the general procedure described in **2.2** using *N*-(phenylseleno)phthalimide as a selenation agent and a reaction time of 3 h. It was obtained as a colourless solid (8.4 mg, 20 μ mol, 40% yield, e.r. = 74:26). **TLC** (heptanes:EtOAc = 3.5:1): R_f = 0.51 (UV). $[\alpha]_D^{23}$ = -61.3 (*c* 0.53, CHCl₃, e.r. = 74:26). **¹H-NMR** (300 MHz, CDCl₃, 298.0 K):

δ / ppm = 7.92-7.89 (m, 2H), 7.58-7.52 (m, 1H), 7.47-7.42 (m, 2H), 7.32-7.15 (m, 10H), 4.07 (d, J = 11.2 Hz, 1H), 4.01 (d, J = 11.2 Hz, 1H), 3.56 (d, J = 13.8 Hz, 1H), 3.43 (d, J = 13.8 Hz, 1H). **¹³C-NMR** (75 MHz, CDCl₃, 298.0 K): δ / ppm = 177.0, 161.1, 136.4, 134.3, 133.2, 130.3, 129.5, 128.9, 128.8, 128.5, 128.2, 127.6, 127.4, 125.3, 69.4, 42.7, 28.7. **HRMS** (ESI-QTOF, MeOH) m/z : $[M+H]^+$ calculated for C₂₃H₂₀NO₂Se, 422.0654; found, 422.0652. **HPLC**: Chiralpak AD-H (*n*-hexane:*i*-PrOH = 20:1, flow rate 0.3 mL/min, 10 °C, λ = 220 nm), retention times t_R (minor) = 39.3 min, t_R (major) = 45.9 min.

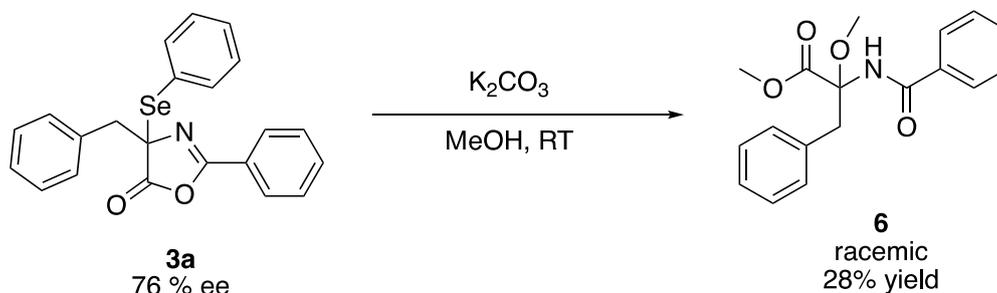
***tert*-Butyl- ((1*S*)-1-(4-benzyl-5-oxo-4-(phenylselanyl)-4,5-dihydrooxazol-2-yl)ethyl)carbamate (8):**

Compound **8** was prepared according to the general procedure described in **2.2** using *N*-(phenylseleno)phthalimide as a selenation agent and quinidine **QD** as a catalyst. It was obtained as a colourless solid (20.4 mg, 43 μ mol, 86% yield, d.r. = 17:83). The two diastereomers were separated using preparative HPLC.

TLC (heptanes:EtOAc = 3.5:1): R_f = 0.67 (UV). **¹H-NMR** (300 MHz, CDCl₃, 298.0 K, major diastereomer): δ / ppm = 7.60 (d, J = 7.1 Hz, 2H), 7.47-7.41 (m, 1H), 7.36-7.31 (m, 2H), 7.24-7.15 (m, 5H), 4.66 (d, J = 8.1 Hz, 1H), 4.10-4.05 (m, 1H), 3.51-3.41 (m, 2H), 1.46 (s, 9H), 0.89 (d, J = 6.9 Hz, 3H). **¹H-NMR** (300 MHz, CDCl₃, 298.0 K, minor diastereomer): δ / ppm = 7.64-7.61 (m, 2H), 7.44-7.38 (m, 1H), 7.35-7.30 (m, 2H), 7.25-7.23 (m, 3H), 7.19-7.15 (m, 2H), 4.66 (d, J = 7.9 Hz, 1H), 4.23-4.11 (m, 1H), 3.48 (d, J = 13.6 Hz, 1H), 3.39 (d, J = 13.6 Hz, 1H), 1.45 (s, 9H), 0.82 (d, J = 7.1 Hz, 3H). **HRMS** (ESI-QTOF, MeOH) m/z : $[M+H]^+$ calculated for C₂₃H₂₇N₂O₄Se, 475.1131; found, 475.1126. **Preparative HPLC**: Grace Alltima Silica 10 μ m, 250 x 10 mm (*n*-hexane:EtOAc = 9:1, flow rate 5 mL/min, λ = 230 nm), retention times t_R (major) = 12.3 min, t_R (minor) = 14.4 min.

4. Further Transformations of 4-Benzyl-2-phenyl-4-(phenylselanyl)oxazol-5(4H)-one **3a**

Methyl 2-benzamido-2-methoxy-3-phenylpropanoate (**6**):

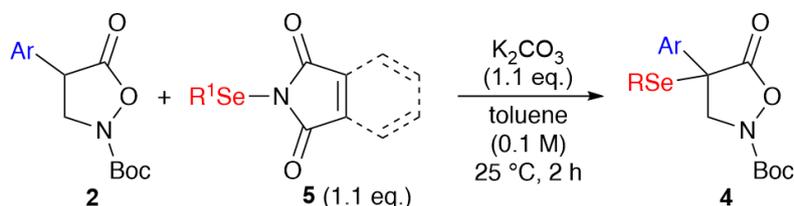


Compound **6** was prepared in analogy to a reported procedure⁴ by addition of a solution of 4-benzyl-2-phenyl-4-(phenylselanyl)oxazol-5(4H)-one (**3a**, 0.046 mmol, 18.6 mg, 1 equiv.) in 0.115 mL methanol to a suspension of K_2CO_3 (0.046 mmol, 6.4 mg, 1 equiv.) in 0.115 mL methanol (in total 0.2 M concerning the substrate **3a**). The resulting mixture was stirred at room temperature under an argon atmosphere for 21 h. After extractive work up using DCM and sat. NaCl solution (aq.) the organic phases were dried over Na_2SO_4 and the solvent was evaporated to obtain 10.5 mg of crude product **6** (28 % yield, e.r. = 52.5:47.5). **TLC** (heptanes:EtOAc = 4:1): R_f = 0.24 (UV). **$^1\text{H-NMR}$** (300 MHz, CDCl_3 , 298.0 K): δ / ppm = 7.74-7.71 (m, 2H), 7.56-7.50 (m, 1H), 7.46-7.41 (m, 2H), 7.27-7.24 (m, 2H), 7.17-7.10 (m, 3H), 4.06 (d, J = 13.4 Hz, 1H), 3.88 (s, 3H), 3.33 (d, J = 13.2 Hz, 1H), 3.31 (s, 3H). **$^{13}\text{C-NMR}$** (75 MHz, CDCl_3 , 298.0 K): δ / ppm = 170.8, 166.9, 134.5, 134.3, 132.2, 130.3, 128.9, 128.6, 127.5, 127.2, 89.2, 53.3, 52.3, 41.2. **HRMS** (ESI-QTOF, MeOH) m/z : $[\text{M}+\text{Na}]^+$ calculated for $\text{C}_{18}\text{H}_{19}\text{NO}_4\text{Na}$, 336.1206; found, 336.1207. **HPLC**: Chiralpak AD-H (n -hexane: i -PrOH = 20:1, flow rate 0.9 mL/min, 10 °C, λ = 220 nm), retention times $t_{\text{R}}(\text{major})$ = 41.7 min, $t_{\text{R}}(\text{minor})$ = 43.6 min.

⁴ J. Yang, W. Sun, Z. He, C. Yu, G. Bao, Y. Li, Y. Liu, L. Hong and R. Wang, *Org. Lett.* 2018, **20**, 22, 7080-7084

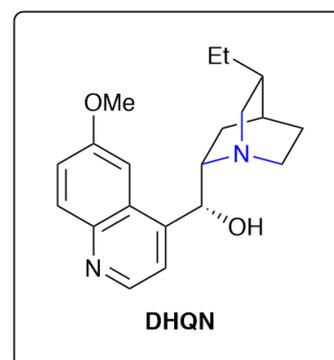
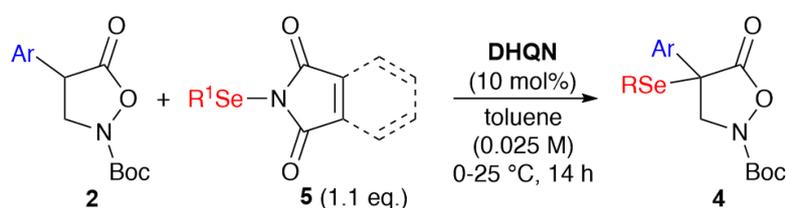
5. Synthesis of α -Selenated α -Aryl-Isoxazolidin-5-ones 4

5.1 General Racemic Procedure



A flame-dried Schlenk-tube equipped with a stirring bar under argon atmosphere was charged with α -Aryl-Isoxazolidin-5-one **2** (0.1 mmol, 1 equiv), K_2CO_3 (15.2 mg, 1.1 equiv) and dry toluene (1 mL, 0.1 M with respect to **2**). Selenation reagent **5** (1.1-1.2 equiv) was added and the reaction flask was covered with aluminum foil. The reaction mixture was stirred for 2 h, whereupon it was concentrated under reduced pressure and directly subjected to column chromatography with gradient elution (silica gel, heptanes/EtOAc = 1/0 to 10/1) to obtain alpha-selenation products **4**.

5.2 General Enantioselective Procedure

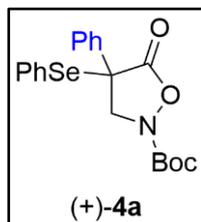


A flame-dried Schlenk-tube equipped with a stirring bar under argon atmosphere was charged with α -Aryl-Isoxazolidin-5-one **2** (1 equiv), **DHQN** (10 mol%) and dry toluene (0.025 M with respect to **2**). The mixture was stirred until all solids dissolved to give a clear, colorless solution which was cooled in an ice bath for 10 min. Selenation reagent **5** (1.1-1.2 equiv) was added at once and the reaction flask was covered with aluminum foil. The reaction mixture was gradually warmed to room temperature and stirred for 14 h, whereupon it was concentrated under reduced pressure and directly subjected to column chromatography with gradient elution (silica gel, heptanes/EtOAc = 1/0 to 10/1) to obtain alpha-selenation products **4** in the given yields and enantiopurities.

6. Characterization Data of α -Selenated α -Aryl-Isoxazolidin-5-ones 4

tert-Butyl 5-oxo-4-phenyl-4-(phenylselenanyl)isoxazolidine-2-carboxylate (**4a**):

Compound (+)-**4a** was prepared from **2a** (199 mg, 756 μ mol) and *N*-(phenylseleno)succinimide



9 (207 mg, 1.1 equiv) according to the general procedure described in **5.2** and was obtained as a colorless oil, which solidifies upon storage in a refrigerator.

(227 mg, 543 μ mol, 72% yield, e.r. = 83:17). **TLC** (heptanes:EtOAc = 10:1):

R_f = 0.24 (UV). $[\alpha]_D^{23}$ = +90.2 (*c* 1.01, CHCl₃). **¹H-NMR** (300 MHz, CDCl₃,

298.0 K): δ / ppm = 7.41-7.34 (m, 3H), 7.30-7.18 (m, 7H), 4.75 (d, *J* =

12.7 Hz, 1H), 4.34 (d, *J* = 12.7 Hz, 1H), 1.56 (s, 9H). **¹³C-NMR** (75 MHz, CDCl₃, 298.0 K): δ

/ ppm = 172.1, 156.6, 138.3, 135.7, 130.6, 129.3, 128.8, 128.6, 127.5, 126.3, 84.7, 59.5, 49.3,

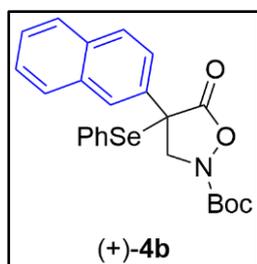
28.4. **HRMS** (ESI-QTOF, MeOH) *m/z*: [M+NH₄]⁺ calculated for C₂₀H₂₅N₂O₄Se⁺, 437.0974;

found, 437.0973. **HPLC**: Chiralpak AD-H (*n*-hexane:*i*-PrOH = 20:1, flow rate 1.0 mL/min,

20 °C, λ = 254 nm), retention times t_R (minor) = 9.7 min, t_R (major) = 11.0 min.

tert-Butyl 4-(naphthalen-2-yl)-5-oxo-4-(phenylselenanyl)isoxazolidine-2-carboxylate (**4b**):

Compound (+)-**4b** was prepared from **2b** (31.8 mg, 101 μ mol) and



N-(phenylseleno)succinimide **9** (28 mg, 1.1 equiv) according to the general procedure described in **5.2** and was obtained as a colorless oil

(31.2 mg, 67 μ mol, 66% yield, e.r. = 82:18). **TLC**

(heptanes:EtOAc = 10:1): R_f = 0.23 (UV). $[\alpha]_D^{23}$ = +84.6 (*c* 1.02,

CHCl₃). **¹H-NMR** (300 MHz, CDCl₃, 298.0 K): δ / ppm = 7.85-7.78 (m,

2H), 7.68-7.60 (m, 3H), 7.53-7.43 (m, 2H), 7.37-7.30 (m, 1H), 7.22-7.10 (m, 4H), 4.85 (d, *J* =

12.7 Hz, 1H), 4.43 (d, *J* = 12.7 Hz, 1H), 1.57 (s, 9H). **¹³C-NMR** (75 MHz, CDCl₃, 298.0 K): δ

/ ppm = 172.0, 156.7, 138.3, 132.9, 132.6, 130.6, 129.3, 128.9, 128.7, 127.8, 127.2, 126.9,

126.7, 126.4, 124.9, 84.8, 59.5, 49.7, 28.4. **HRMS** (ESI-QTOF, MeOH) *m/z*: [M+NH₄]⁺

calculated for C₂₄H₂₇N₂O₄Se⁺, 487.1131; found, 487.1129. **HPLC**: Chiralpak AD-H

(*n*-hexane:*i*-PrOH = 20:1, flow rate 1.0 mL/min, 20 °C, λ = 254 nm), retention times

t_R (minor) = 12.5 min, t_R (major) = 16.7 min.

***tert*-Butyl 5-oxo-4-(phenylselanyl)-4-(thiophen-3-yl)isoxazolidine-2-carboxylate (4c):**

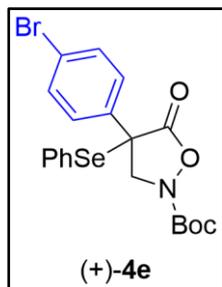
Compound (+)-**4c** was prepared from **2c** (26.8 mg, 100 μ mol) and *N*-(phenylseleno)succinimide **9** (28 mg, 1.1 equiv) according to the general procedure described in **5.2** and was obtained as a yellowish oil (21.6 mg, 51 μ mol, 51% yield, e.r. = 76:24). **TLC** (heptanes:EtOAc = 10:1): R_f = 0.30 (UV). $[\alpha]_D^{23}$ = +54.7 (*c* 0.98, CHCl₃). **¹H-NMR** (300 MHz, CDCl₃, 298.0 K): δ / ppm = 7.43-7.36 (m, 1H), 7.33 (dd, *J* = 5.1, 3.0 Hz, 1H), 7.28-7.21 (m, 4H), 7.17 (dd, *J* = 5.1, 1.4 Hz, 1H), 7.05 (dd, *J* = 3.0, 1.4 Hz, 1H), 4.74 (d, *J* = 12.6 Hz, 1H), 4.27 (d, *J* = 12.6 Hz, 1H), 1.58 (s, 9H). **¹³C-NMR** (75 MHz, CDCl₃, 298.0 K): δ / ppm = 172.1, 156.7, 138.3, 135.2, 130.6, 129.3, 126.8, 126.4, 124.2, 84.8, 59.6, 46.7, 28.5. **HRMS** (ESI-QTOF, MeOH) *m/z*: [M+NH₄]⁺ calculated for C₁₈H₂₃N₂O₄SSe⁺, 443.0538; found, 443.0538. **HPLC**: Chiralpak AD-H (*n*-hexane:*i*-PrOH = 20:1, flow rate 1.0 mL/min, 20 °C, λ = 254 nm), retention times t_R (minor) = 14.1 min, t_R (major) = 15.7 min.

***tert*-Butyl 4-(4-chlorophenyl)-5-oxo-4-(phenylselanyl)isoxazolidine-2-carboxylate (4d):**

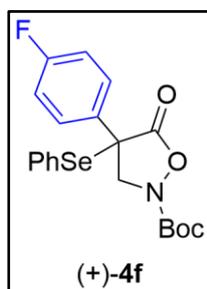
Compound (+)-**4d** was prepared from **2d** (30.6 mg, 103 μ mol) and *N*-(phenylseleno)succinimide **9** (28 mg, 1.1 equiv) according to the general procedure described in **5.2** and was obtained as a colorless oil (34.8 mg, 77 μ mol, 75% yield, e.r. = 80:20). **TLC** (heptanes:EtOAc = 10:1): R_f = 0.24 (UV). $[\alpha]_D^{24}$ = +93.3 (*c* 1.01, CHCl₃). **¹H-NMR** (300 MHz, CDCl₃, 298.0 K): δ / ppm = 7.45-7.36 (m, 1H), 7.32-7.21 (m, 8H), 4.77 (d, *J* = 12.7 Hz, 1H), 4.27 (d, *J* = 12.7 Hz, 1H), 1.58 (s, 9H). **¹³C-NMR** (75 MHz, CDCl₃, 298.0 K): δ / ppm = 171.8, 156.6, 138.3, 134.7, 134.4, 130.9, 129.5, 128.9, 128.8, 126.0, 84.9, 59.2, 48.4, 28.4. **HRMS** (ESI-QTOF, MeOH) *m/z*: [M+NH₄]⁺ calculated for C₂₀H₂₄ClN₂O₄Se⁺, 471.0584; found, 471.0582. **HPLC**: Chiralpak AD-H (*n*-hexane:*i*-PrOH = 20:1, flow rate 1.0 mL/min, 20 °C, λ = 254 nm), retention times t_R (minor) = 12.0 min, t_R (major) = 16.1 min.

***tert*-Butyl 4-(4-bromophenyl)-5-oxo-4-(phenylselanyl)isoxazolidine-2-carboxylate (4e):**

Compound (+)-**4e** was prepared from **2e** (34.6 mg, 101 μ mol) and *N*-(phenylseleno)succinimide **9** (28 mg, 1.1 equiv) according to the general procedure described in **5.2** and was obtained as a colorless oil (38.1 mg, 77 μ mol, 76% yield, e.r. = 83:17). **TLC** (heptanes:EtOAc = 10:1): R_f = 0.27 (UV). $[\alpha]_D^{24}$ = +93.3 (c 1.00, CHCl₃). **¹H-NMR** (300 MHz, CDCl₃, 298.0 K): δ / ppm = 7.44-7.36 (m, 3H), 7.28-7.20 (m, 6H), 4.76 (d, J = 12.7 Hz, 1H), 4.26 (d, J = 12.7 Hz, 1H), 1.58 (s, 9H). **¹³C-NMR** (75 MHz, CDCl₃, 298.0 K): δ / ppm = 171.7, 156.5, 138.3, 134.9, 131.9, 130.9, 129.5, 129.0, 126.0, 122.9, 84.9, 59.2, 48.5, 28.4. **HRMS** (ESI-QTOF, MeOH) m/z : [M+NH₄]⁺ calculated for C₂₀H₂₄BrN₂O₄Se⁺, 515.0079; found, 515.0079. **HPLC**: Chiralpak AD-H (*n*-hexane:*i*-PrOH = 20:1, flow rate 1.0 mL/min, 20 °C, λ = 254 nm), retention times t_R (minor) = 12.6 min, t_R (major) = 17.6 min.

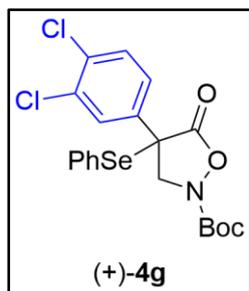
***tert*-Butyl 4-(4-fluorophenyl)-5-oxo-4-(phenylselanyl)isoxazolidine-2-carboxylate (4f):**

Compound (+)-**4f** was prepared from **2f** (28.4 mg, 101 μ mol) and *N*-(phenylseleno)succinimide **9** (28 mg, 1.1 equiv) according to the general procedure described in **5.2** and was obtained as a colorless oil (29.8 mg, 68 μ mol, 68% yield, e.r. = 81:19). **TLC** (heptanes:EtOAc = 10:1): R_f = 0.24 (UV). $[\alpha]_D^{23}$ = +78.4 (c 0.99, CHCl₃). **¹H-NMR** (300 MHz, CDCl₃, 298.0 K): δ / ppm = 7.43-7.31 (m, 3H), 7.27-7.23 (m, 4H), 6.99-6.91 (m, 2H), 4.77 (d, J = 12.7 Hz, 1H), 4.29 (d, J = 12.7 Hz, 1H), 1.58 (s, 9H). **¹³C-NMR** (75 MHz, CDCl₃, 298.0 K): δ / ppm = 172.0, 162.6 (d, $^1J_{CF}$ = 249.0 Hz), 156.6, 138.3, 131.7 (d, $^3J_{CF}$ = 3.4 Hz), 130.8, 129.4, 129.4 (d, $^3J_{CF}$ = 8.0 Hz), 126.1, 115.7 (d, $^2J_{CF}$ = 21.6 Hz), 84.9, 59.5, 48.5, 28.5. **¹⁹F-NMR** (282 MHz, CDCl₃, 298.0 K): δ / ppm = -113.1. **HRMS** (ESI-QTOF, MeOH) m/z : [M+NH₄]⁺ calculated for C₂₀H₂₄FN₂O₄Se⁺, 455.0880; found, 455.0881. **HPLC**: Chiralpak AD-H (*n*-hexane:*i*-PrOH = 20:1, flow rate 1.0 mL/min, 20 °C, λ = 254 nm), retention times t_R (minor) = 12.1 min, t_R (major) = 14.2 min.



***tert*-Butyl 4-(3,4-dichlorophenyl)-5-oxo-4-(phenylselanyl)isoxazolidine-2-carboxylate (4g):**

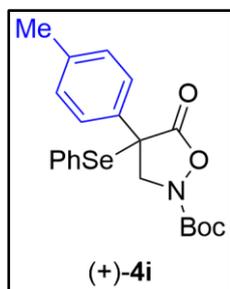
Compound (+)-**4g** was prepared from **2g** (33.0 mg, 99 μ mol) and *N*-(phenylseleno)succinimide



9 (28 mg, 1.1 equiv) according to the general procedure described in **5.2** and was obtained as a colorless oil (34.7 mg, 71 μ mol, 72% yield, e.r. = 75:25). **TLC** (heptanes:EtOAc = 10:1): R_f = 0.30 (UV). $[\alpha]_D^{24}$ = +77.2 (*c* 0.96, CHCl₃). **¹H-NMR** (300 MHz, CDCl₃, 298.0 K): δ / ppm = 7.46-7.40 (m, 1H), 7.37 (d, J = 2.3 Hz, 1H), 7.33 (d, J = 8.5 Hz, 1H), 7.31-7.24 (m, 4H), 7.18 (dd, J = 8.5, 2.3 Hz, 1H), 4.77 (d, J = 12.7 Hz, 1H), 4.23 (d, J = 12.7 Hz, 1H), 1.59 (s, 9H). **¹³C-NMR** (75 MHz, CDCl₃, 298.0 K): δ / ppm = 171.4, 156.5, 138.4, 136.0, 133.0, 132.9, 131.2, 130.6, 129.6, 129.4, 126.6, 125.8, 85.1, 59.0, 47.6, 28.5 **HRMS** (ESI-QTOF, MeOH) m/z : $[M+NH_4]^+$ calculated for C₂₀H₂₃Cl₂N₂O₄Se⁺, 505.0195; found, 505.0195. **HPLC**: Chiralpak AD-H (*n*-hexane:*i*-PrOH = 20:1, flow rate 1.0 mL/min, 20 °C, λ = 254 nm), retention times t_R (minor) = 10.2 min, t_R (major) = 13.1 min.

***tert*-Butyl 5-oxo-4-(phenylselanyl)-4-(*p*-tolyl)isoxazolidine-2-carboxylate (4i):**

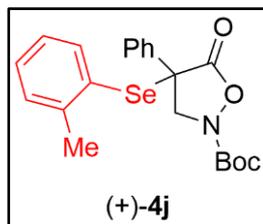
Compound (+)-**4i** was prepared from **2i** (28.4 mg, 102 μ mol) and *N*-(phenylseleno)succinimide



9 (28 mg, 1.1 equiv) according to the general procedure described in **5.2** and was obtained as a reddish oil (18.3 mg, 42 μ mol, 41% yield, e.r. = 74:26). **TLC** (heptanes:EtOAc = 10:1): R_f = 0.27 (UV). $[\alpha]_D^{23}$ = +44.6 (*c* 0.99, CHCl₃). **¹H-NMR** (500 MHz, CDCl₃, 298.0 K): δ / ppm = 7.40-7.36 (m, 1H), 7.30-7.21 (m, 6H), 7.08 (d, J = 8.1 Hz, 2H), 4.71 (d, J = 12.6 Hz, 1H), 4.32 (d, J = 12.6 Hz, 1H), 2.32 (s, 3H), 1.55 (s, 9H). **¹³C-NMR** (126 MHz, CDCl₃, 298.0 K): δ / ppm = 172.2, 156.6, 138.6, 138.3, 132.6, 130.6, 129.5, 129.3, 127.3, 126.4, 84.6, 59.6, 49.5, 28.4, 21.5. **HRMS** (ESI-QTOF, MeOH) m/z : $[M+NH_4]^+$ calculated for C₂₁H₂₇N₂O₄Se⁺, 451.1131; found, 451.1128. **HPLC**: Chiralpak AD-H (*n*-hexane:*i*-PrOH = 20:1, flow rate 1.0 mL/min, 20 °C, λ = 254 nm), retention times t_R (minor) = 9.2 min, t_R (major) = 12.6 min.

***tert*-Butyl 5-oxo-4-phenyl-4-(*o*-tolylselenanyl)isoxazolidine-2-carboxylate (4j):**

Compound (+)-**4j** was prepared from **2a** (26.5 mg, 101 μ mol) and *N*-(*o*-tolylseleno)phthalimide



(38 mg, 1.2 equiv) according to the general procedure described in **5.2**

and was obtained as a colorless oil (37.6 mg, 87 μ mol, 76% yield, e.r. =

76:24). **TLC** (heptanes:EtOAc = 10:1): R_f = 0.32 (UV). $[\alpha]_D^{24} = +79.5$

(c 0.99, CHCl_3). **$^1\text{H-NMR}$** (500 MHz, CDCl_3 , 298.0 K): δ / ppm =

7.36-7.33 (m, 2H), 7.30-7.23 (m, 5H), 7.19 (d, J = 7.4 Hz, 1H), 7.05-7.01

(m, 1H), 4.87 (d, J = 12.7 Hz, 1H), 4.32 (d, J = 12.7 Hz, 1H), 2.07 (s, 3H), 1.58 (s, 9H).

$^{13}\text{C-NMR}$ (126 MHz, CDCl_3 , 298.0 K): δ / ppm = 171.6, 156.7, 144.9, 140.0, 135.8, 131.2,

130.5, 128.8, 128.6, 127.3, 127.2, 126.7, 84.7, 59.8, 48.3, 28.4, 23.0. **HRMS** (ESI-QTOF,

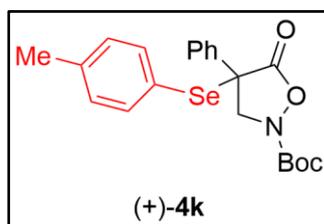
MeOH) m/z : $[\text{M}+\text{NH}_4]^+$ calculated for $\text{C}_{21}\text{H}_{27}\text{N}_2\text{O}_4\text{Se}^+$, 451.1131; found, 451.1125. **HPLC**:

Chiralpak AD-H (*n*-hexane:*i*-PrOH = 20:1, flow rate 1.0 mL/min, 10 $^\circ\text{C}$, λ = 240 nm),

retention times $t_{R(\text{minor})}$ = 9.1 min, $t_{R(\text{major})}$ = 11.8 min.

***tert*-Butyl 5-oxo-4-phenyl-4-(*p*-tolylselenanyl)isoxazolidine-2-carboxylate (4k):**

Compound (+)-**4k** was prepared from **2a** (26.4 mg, 100 μ mol) and



N-(*p*-tolylseleno)phthalimide (38 mg, 1.2 equiv) according to the

general procedure described in **5.2** and was obtained as a colorless

oil (8.3 mg, 19 μ mol, 19% yield, e.r. = 72:28). **TLC**

(heptanes:EtOAc = 10:1): R_f = 0.32 (UV). $[\alpha]_D^{24} = +44.6$ (c 0.83,

CHCl_3). **$^1\text{H-NMR}$** (300 MHz, CDCl_3 , 298.0 K): δ / ppm = 7.41-7.34

(m, 2H), 7.30-7.25 (m, 3H), 7.14 (d, J = 7.9 Hz, 2H), 7.03 (d, J = 7.9 Hz, 2H), 4.73 (d, J =

12.7 Hz, 1H), 4.32 (d, J = 12.7 Hz, 1H), 2.33 (s, 3H), 1.56 (s, 9H). **$^{13}\text{C-NMR}$** (75 MHz, CDCl_3 ,

298.0 K): δ / ppm = 172.1, 156.7, 141.1, 138.2, 135.9, 130.2, 128.8, 128.5, 127.4, 122.9, 84.6,

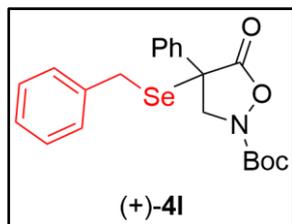
59.4, 49.3, 28.4, 21.7. **HRMS** (ESI-QTOF, MeOH) m/z : $[\text{M}+\text{NH}_4]^+$ calculated for

$\text{C}_{21}\text{H}_{27}\text{N}_2\text{O}_4\text{Se}^+$, 451.1131; found, 451.1129. **HPLC**: Chiralpak AD-H

(*n*-hexane:*i*-PrOH = 20:1, flow rate 1.0 mL/min, 10 $^\circ\text{C}$, λ = 240 nm), retention times

***tert*-Butyl 4-(benzylselenanyl)-5-oxo-4-phenylisoxazolidine-2-carboxylate (**4l**):**

Compound (+)-**4l** was prepared from **2a** (26.6 mg, 101 μ mol) and *N*-(benzylseleno)phthalimide



(38 mg, 1.2 equiv) according to the general procedure described in **5.2**

and was obtained as a colorless oil (9.7 mg, 22 μ mol, 22% yield, e.r. = 61:39). **TLC** (heptanes:EtOAc = 10:1): R_f = 0.32 (UV). $[\alpha]_D^{23}$ = +17.3

(*c* 0.97, CHCl₃). **¹H-NMR** (500 MHz, CDCl₃, 298.0 K): δ / ppm = 7.70-7.67 (m, 2H), 7.42-7.38 (m, 2H), 7.36-7.32 (m, 1H), 7.22-7.15 (m,

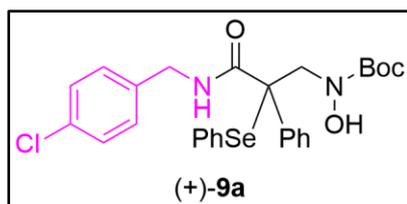
3H), 7.11-7.08 (m, 2H), 4.65 (d, J = 12.7 Hz, 1H), 4.18 (d, J = 12.7 Hz, 1H), 4.04 (d, J = 10.8 Hz, 1H), 3.69 (d, J = 10.8 Hz, 1H), 1.51 (s, 9H). **¹³C-NMR** (126 MHz, CDCl₃, 298.0 K): δ / ppm = 172.8, 156.5, 136.2, 135.7, 129.6, 129.3, 128.9, 127.8, 127.6, 84.8, 61.5, 47.2, 30.5,

28.4. **HRMS** (ESI-QTOF, MeOH) m/z : $[M+NH_4]^+$ calculated for C₂₁H₂₇N₂O₄Se⁺, 451.1131; found, 451.1133. **HPLC**: Chiralpak OD-H (*n*-hexane:*i*-PrOH = 20:1, flow rate 1.0 mL/min, 10 °C, λ = 220 nm), retention times t_R (minor) = 11.9 min, t_R (major) = 14.1 min.

7. Further Transformations of *N*-Boc 4,4-phenyl(phenylselenanyl)isoxazolidin-5-one 4a

tert-Butyl (3-((4-chlorobenzyl)amino)-3-oxo-2-phenyl-2-(phenylselenanyl)propyl)(hydroxy)carbamate (**9a**):

Compound (+)-**9a** was prepared in analogy to a reported procedure⁵ by treatment of

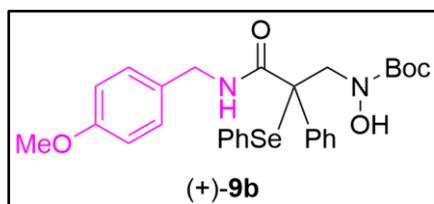


enantioenriched selenation product (+)-**4a** (38.4 mg, 92 μ mol, e.r. = 83:17) with 4-chlorobenzylamine (60 μ L, 5 equiv) in *t*BuOH (2 mL, 0.05 M). The crude product was purified *via* flash column chromatography (silica gel, heptanes/EtOAc =

2/1) to obtain amide (+)-**9a** as a white solid (42.8 mg, 76 μ mol, 83% yield, e.r. = 83:17). **TLC** (heptanes:EtOAc = 2:1): R_f = 0.44 (UV). $[\alpha]_D^{23}$ = +33.2 (*c* 1.04, CHCl₃). **¹H-NMR** (300 MHz, CDCl₃, 298.0 K): δ / ppm = 8.12 (s, 1H), 7.40-7.15 (m, 15H), 6.83 (bt, *J* = 5.7 Hz, 1H), 4.57 (d, *J* = 15.2 Hz, 1H), 4.39 (d, *J* = 6.0 Hz, 2H), 4.07 (d, *J* = 15.2 Hz, 1H), 1.27 (s, 9H). **¹³C-NMR** (75 MHz, CDCl₃, 298.0 K): δ / ppm = 173.9, 155.3, 138.6, 137.5, 136.2, 133.9, 130.0, 129.5, 129.2, 128.8, 128.7, 128.4, 126.8, 81.6, 61.9, 59.1, 44.3, 28.4. **HRMS** (ESI-QTOF, MeOH) *m/z*: [M+Na]⁺ calculated for C₂₇H₂₉ClN₂NaO₄Se⁺, 583.0873; found, 583.0875. **HPLC**: Chiralpak OD-H (*n*-hexane:*i*-PrOH = 2:1, flow rate 0.5 mL/min, 20 °C, λ = 254 nm), retention times t_R (minor) = 13.8 min, t_R (major) = 27.4 min.

tert-Butyl hydroxy(3-((4-methoxybenzyl)amino)-3-oxo-2-phenyl-2-(phenylselenanyl)propyl)carbamate (**9b**):

Analogously prepared to **9a**, using 4-methoxybenzylamine (60 μ L, 5 equiv). Amide (+)-**9b** was



obtained as an off-white solid (42.8 mg, 77 μ mol, 84% yield, e.r. = 83:17). **TLC** (heptanes:EtOAc = 2:1): R_f = 0.37 (UV). $[\alpha]_D^{23}$ = +29.4 (*c* 1.07, CHCl₃). **¹H-NMR**

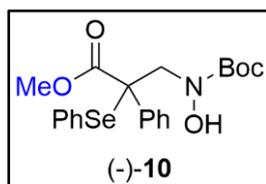
(500 MHz, CDCl₃, 298.0 K): δ / ppm = 8.40 (s, 1H), 7.42-7.39 (m, 2H), 7.38-7.33 (m, 3H), 7.29-7.24 (m, 4H), 7.23-7.16 (m, 4H), 6.88-6.84 (m, 2H), 6.79 (bs, 1H), 4.59 (d, *J* = 15.2 Hz, 1H), 4.38 (dd, *J* = 5.6, 2.4 Hz, 2H), 4.02 (d, *J* = 15.2 Hz, 1H), 3.81 (s, 3H), 1.26 (s, 9H). **¹³C-NMR** (126 MHz, CDCl₃, 298.0 K): δ / ppm = 173.9, 159.6, 155.0, 138.6, 137.5, 130.0, 129.5, 128.7, 128.3, 126.8, 114.5, 81.4, 62.0, 59.4, 55.7, 44.5, 28.4. **HRMS** (ESI-QTOF, MeOH) *m/z*: [M+Na]⁺ calculated for C₂₈H₃₂N₂NaO₅Se⁺, 579.1369; found,

⁵ P. Zebrowski, I. Eder, A. Eitzinger, S. C. Mallojjala and M. Waser, *ACS Org. Inorg. Au* 10.1021/acsorginorgau.1c00025.

579.1364. **HPLC**: Chiralpak OD-H (*n*-hexane:*i*-PrOH = 2:1, flow rate 0.5 mL/min, 20 °C, λ = 254 nm), retention times $t_{\text{R}}(\text{minor})$ = 19.1 min, $t_{\text{R}}(\text{major})$ = 31.3 min.

Methyl 3-((*tert*-butoxycarbonyl)(hydroxy)amino)-2-phenyl-2-(phenylselenanyl)propanoate (10):

A reaction vial was charged with (+)-**4a** (42.4 mg, 101 μmol , e.r. = 83:17), MeOH (1 mL,

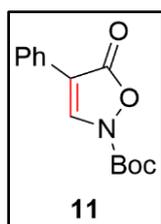


0.1 M) and Amberlyst A21 (100 mg). The heterogenous mixture was stirred vigorously for 24 h at room temperature, whereupon it was filtered over cotton and concentrated under reduced pressure. The crude product was purified by preparative TLC (silica gel, heptanes/EtOAc =

2/1) to obtain (-)-**10** as a colorless oil (21.0 mg, 47 μmol , 46% yield, e.r. = 83:17). **TLC** (heptanes:EtOAc = 2:1): R_f = 0.29 (UV). $[\alpha]_{\text{D}}^{24}$ = -21.1 (*c* 0.43, CHCl_3). **$^1\text{H-NMR}$** (300 MHz, CDCl_3 , 298.0 K): δ / ppm = 7.35-7.29 (m, 3H), 7.25-7.14 (m, 7H), 6.41 (s, 1H), 4.43 (d, J = 14.8 Hz, 1H), 4.25 (d, J = 14.8 Hz, 1H), 3.70 (s, 3H), 1.38 (s, 9H). **$^{13}\text{C-NMR}$** (176 MHz, CDCl_3 , 298.0 K): δ / ppm = 173.6, 156.0, 138.3, 129.8, 129.0, 128.4, 128.3, 127.9, 127.1, 82.3, 60.3, 57.0, 53.3, 28.4. **HRMS** (ESI-QTOF, MeOH) m/z : $[\text{M}+\text{Na}]^+$ calculated for $\text{C}_{21}\text{H}_{25}\text{NNaO}_5\text{Se}^+$, 474.0790; found, 474.0795. **HPLC**: YMC Chiral ART Cellulose-SB (*n*-hexane:*i*-PrOH = 4:1, flow rate 0.5 mL/min, 20 °C, λ = 254 nm), retention times $t_{\text{R}}(\text{major})$ = 22.7 min, $t_{\text{R}}(\text{minor})$ = 26.2 min.

***tert*-Butyl 5-oxo-4-phenylisoxazole-2(5*H*)-carboxylate (11):**

A flame-dried Schlenk tube was charged with **4a** (40.8 mg, 98 μmol) and dry CH_2Cl_2 (1 mL,



0.1 M). mCPBA (49.6 mg, 2.3 equiv, $\leq 77\%$) was added and the mixture was stirred for 24 h at room temperature. The reaction was quenched by addition of sat. $\text{Na}_2\text{S}_2\text{O}_3$ solution (1 mL) and the organic phase was washed with sat. $\text{Na}_2\text{S}_2\text{O}_3$ (2x 1 mL) and sat. NaHCO_3 (3x 1 mL) solution, dried over anhydrous Na_2SO_4 , filtered and concentrated under reduced pressure. **11** was obtained as a off-white

solid (21.2 mg, 81 μmol , 83% yield). **TLC** (heptanes:EtOAc = 5:1): R_f = 0.34 (UV). **$^1\text{H-NMR}$** (300 MHz, CDCl_3 , 298.0 K): δ / ppm = 8.51 (s, 1H), 7.80-7.75 (m, 2H), 7.45-7.30 (m, 3H), 1.63 (s, 9H). **$^{13}\text{C-NMR}$** (75 MHz, CDCl_3 , 298.0 K): δ / ppm = 166.6, 144.9, 138.9, 129.2, 128.7, 128.0, 126.1, 107.5, 87.5, 28.4. **HRMS** (ESI-QTOF, MeOH) m/z : $[\text{M}+\text{Na}]^+$ calculated for $\text{C}_{14}\text{H}_{15}\text{NNaO}_4^+$, 284.0893; found, 284.0893.

8. Single-Crystal Analysis

Single crystals suitable for single crystal X-ray diffraction were obtained by re-crystallisation from diethyl ether. Single-crystal structure analysis was carried out at room temperature on a Bruker D8 Quest ECO diffractometer with graphite-monochromated MoK α radiation ($\lambda = 0.71073 \text{ \AA}$). The structures were solved by direct methods (SHELXS-97⁶) and refined by full-matrix least-squares on F^2 (SHELXL-2014/7⁷). The H atoms were calculated geometrically, and a riding model was applied in the refinement process. Crystallographic details for (*rac*)-**4a** can be found in Table 1. CCDC 2121570 contain the supplementary crystallographic data. This information can be obtained free of charge via <https://www.ccdc.cam.ac.uk/structures/>

Table 1: X-ray diffraction crystal data, data collection and structure refinement for the compound (*rac*)-**4a**.

Compound	(<i>rac</i>)- 4a
Empirical formula	C ₂₀ H ₂₁ NO ₄ Se
Formula weight (g·mol ⁻¹)	418.34
Crystal system	triclinic
Space group	$P\bar{1}$
Temp (K)	293
a (Å)	9.198(5)
b (Å)	10.355(7)
c (Å)	12.174(7)
α (°)	69.365(16)
β (°)	76.44(2)
γ (°)	64.56(2)
V (Å ³)	975.2(10)
Z	2
ρ_{calc} (g·cm ⁻³)	1.425
Reflns collected	60514
Indep. reflns	3429
Obs. reflns [$I > 2\sigma(I)$]	3176
Param. refin./restr.	238 / 0
Absorption correction	multi-scan
R_1	0.027

⁶ Sheldrick, GM (1997) SHELXS-97, Program for the Solution of Crystal Structures, Göttingen, Germany. See also: Sheldrick, GM (1990) Acta Crystallogr A 46:467.

⁷ Sheldrick, GM (2015) Acta Crystallogr A 71:3.

Compound	(<i>rac</i>)-4a
wR_2	0.075
CCDC	2121570

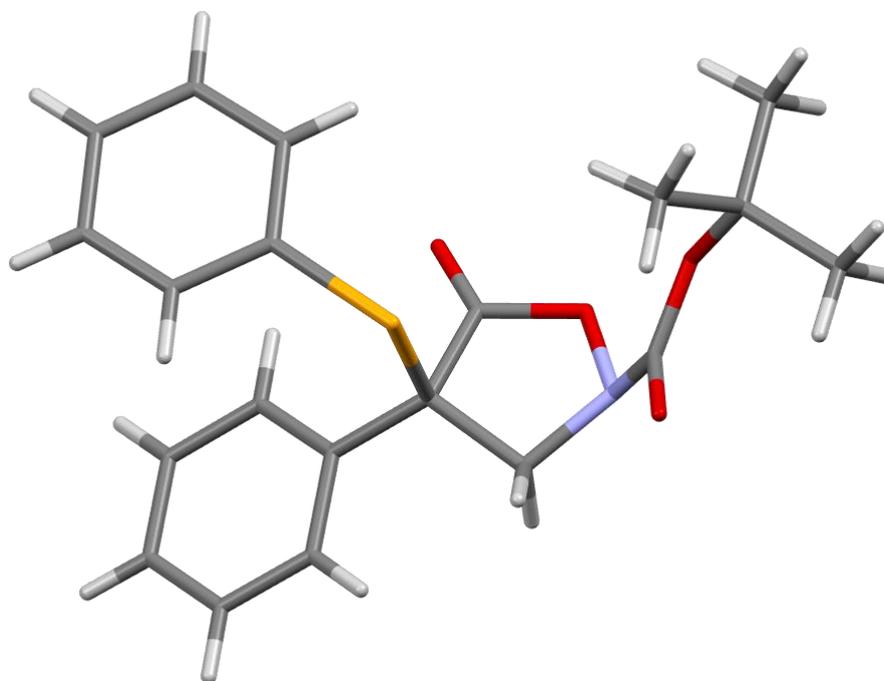
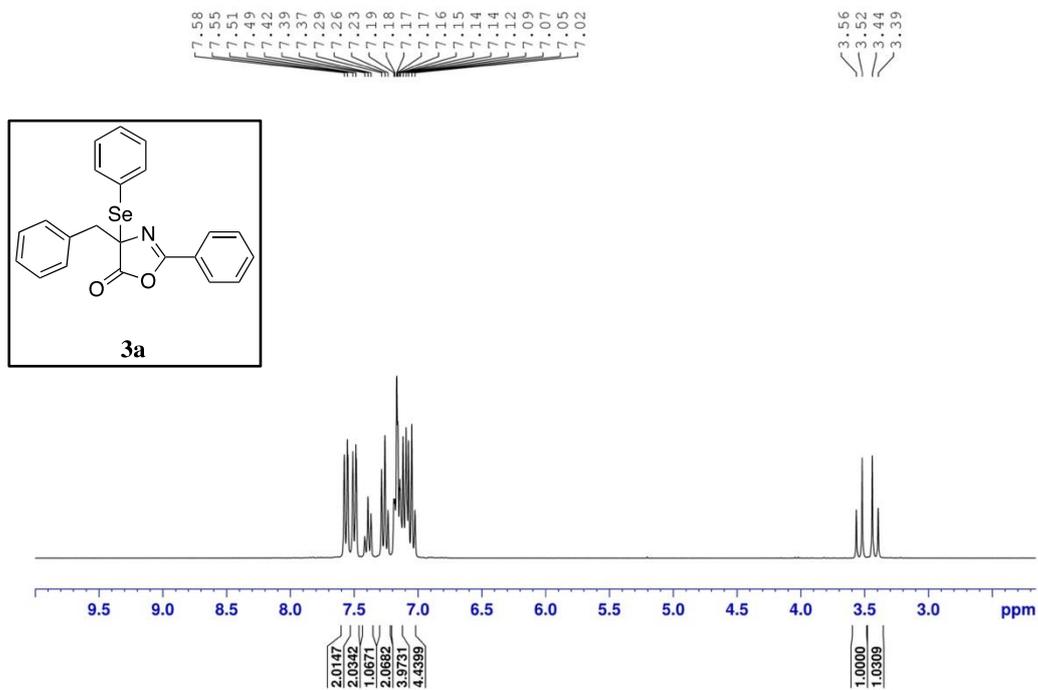


Figure 1: Single crystal structure of (*rac*)-4a.

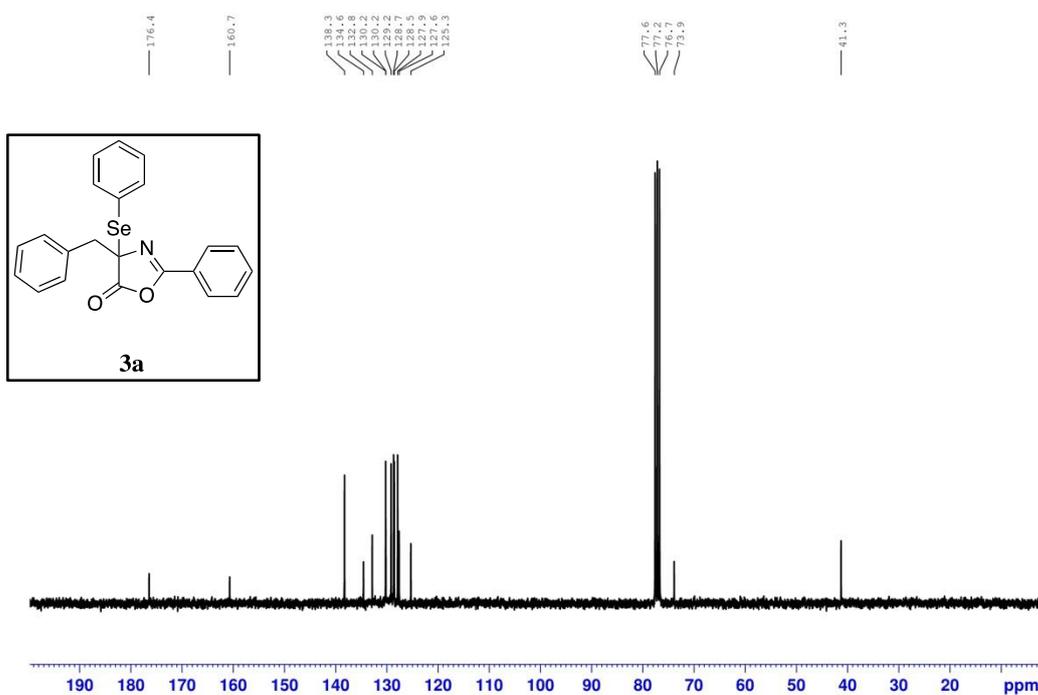
9. NMR Spectra of Selenation Products

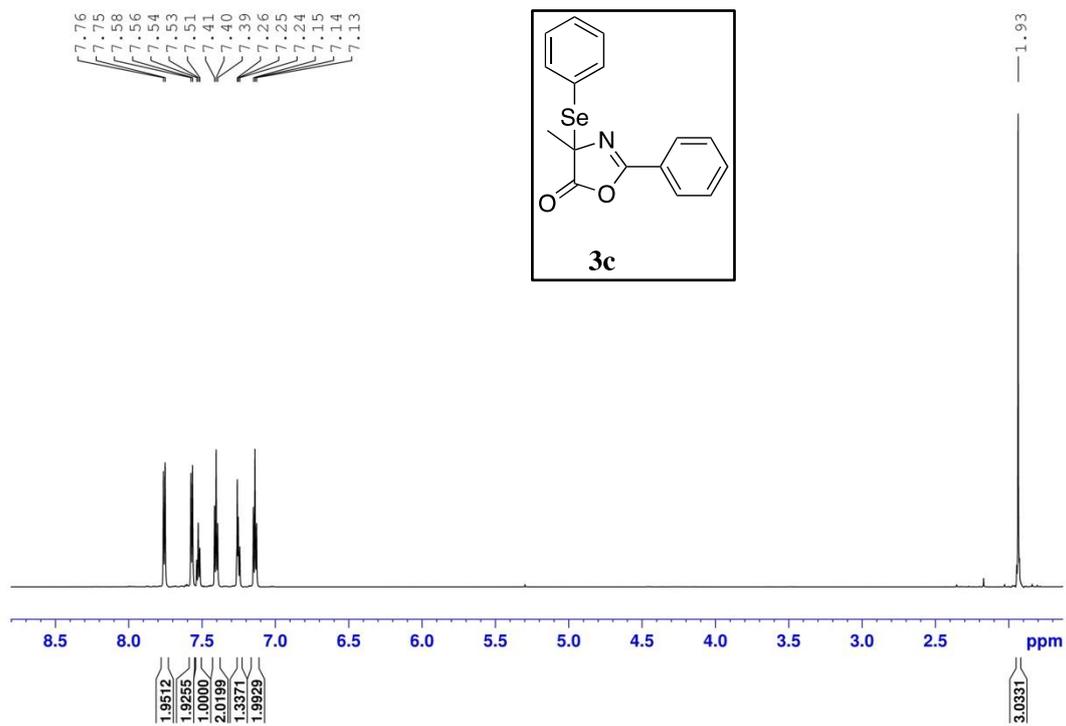
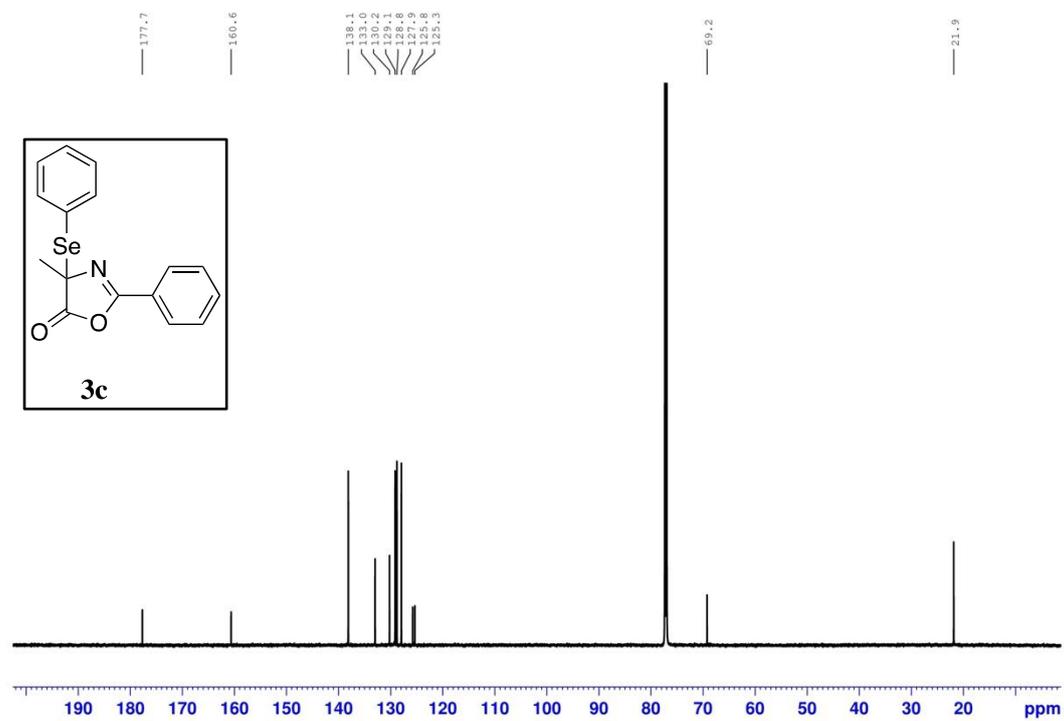
NMR spectra of compound **3a**

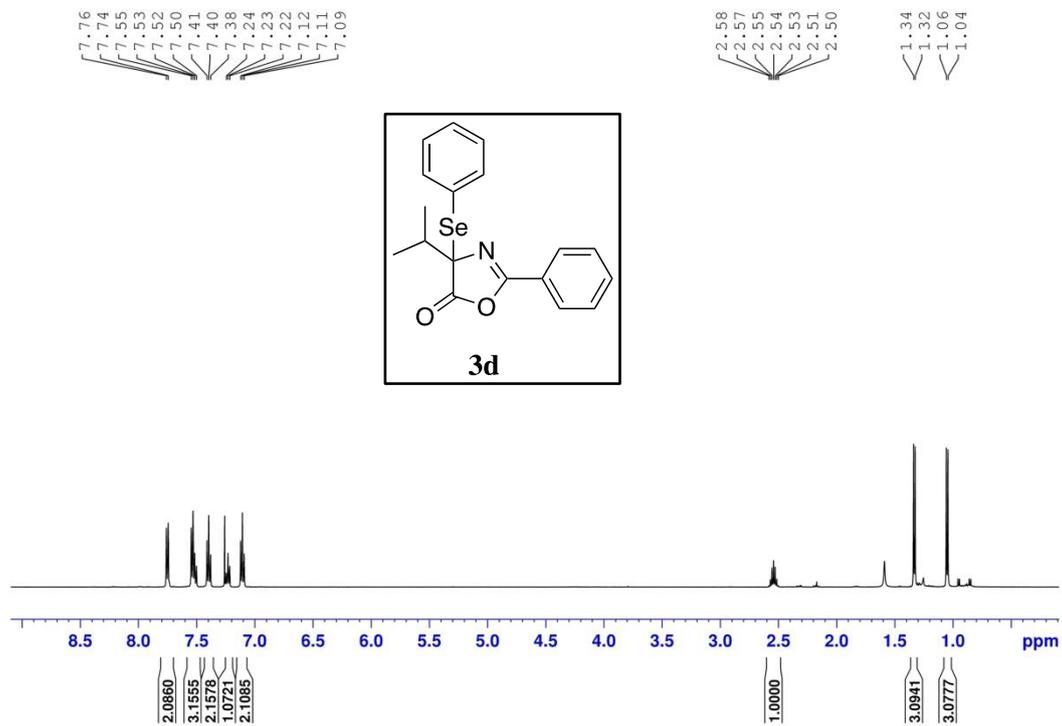
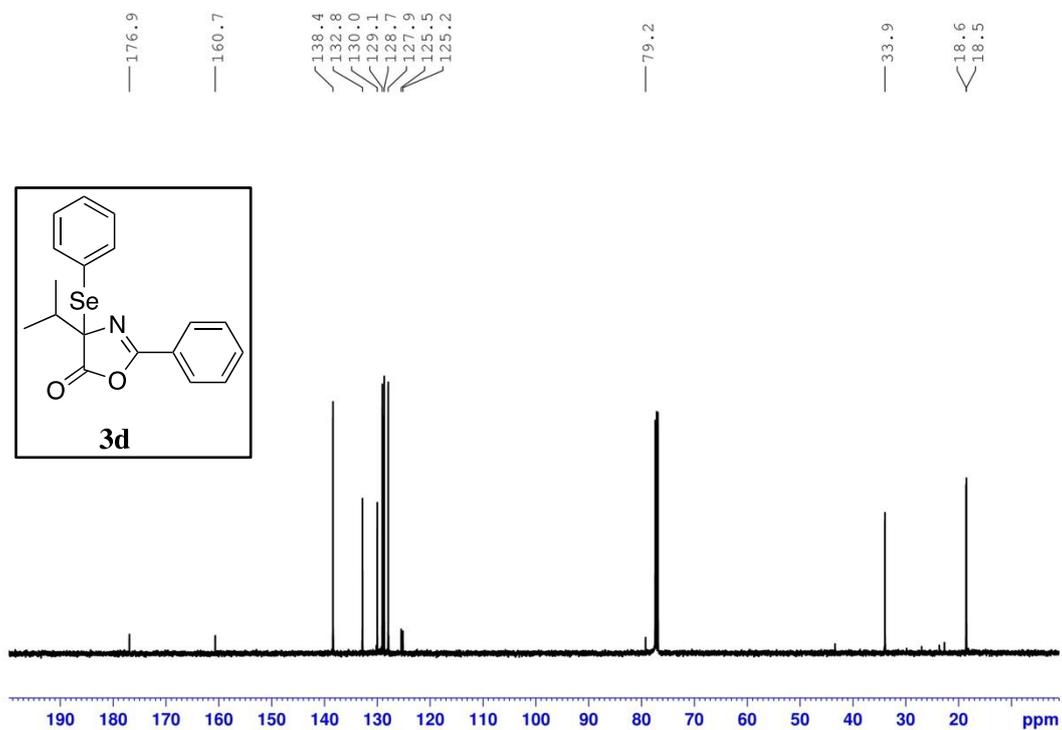
$^1\text{H-NMR}$ (300 MHz, CDCl_3 , 298 K)

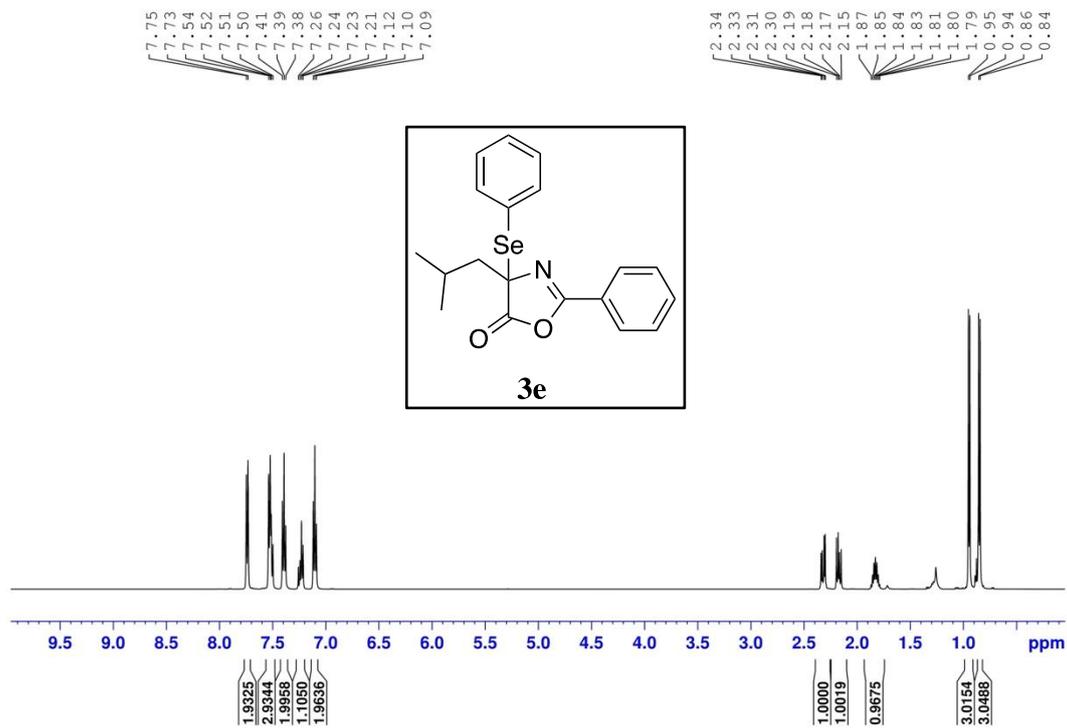
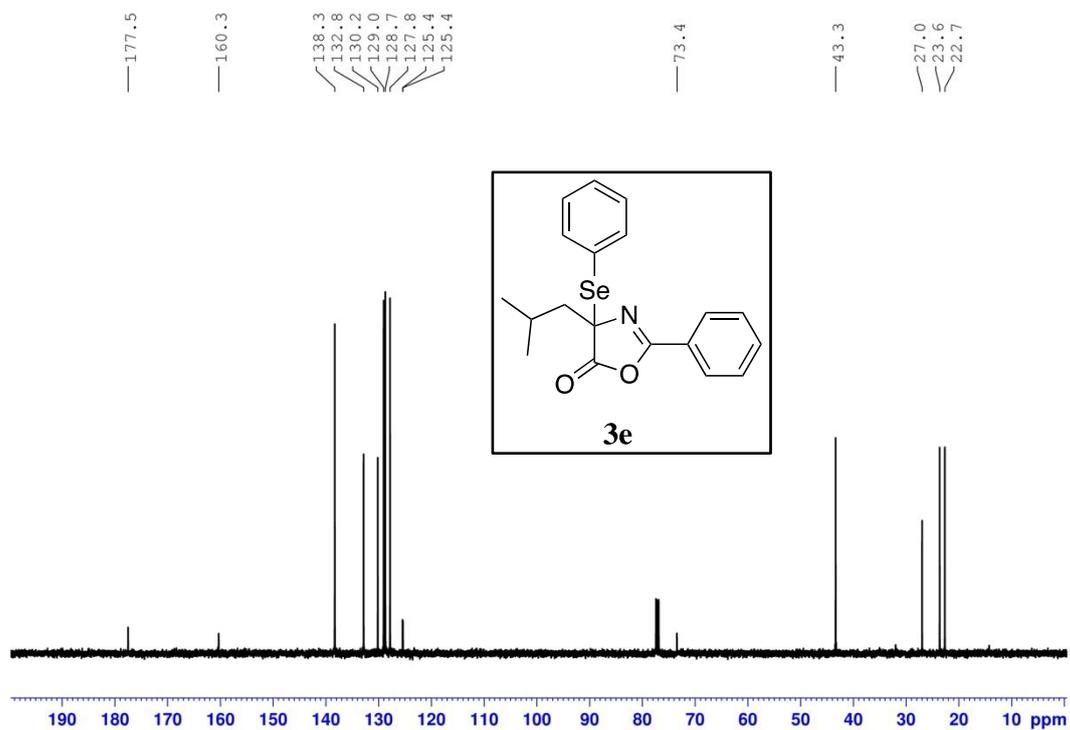


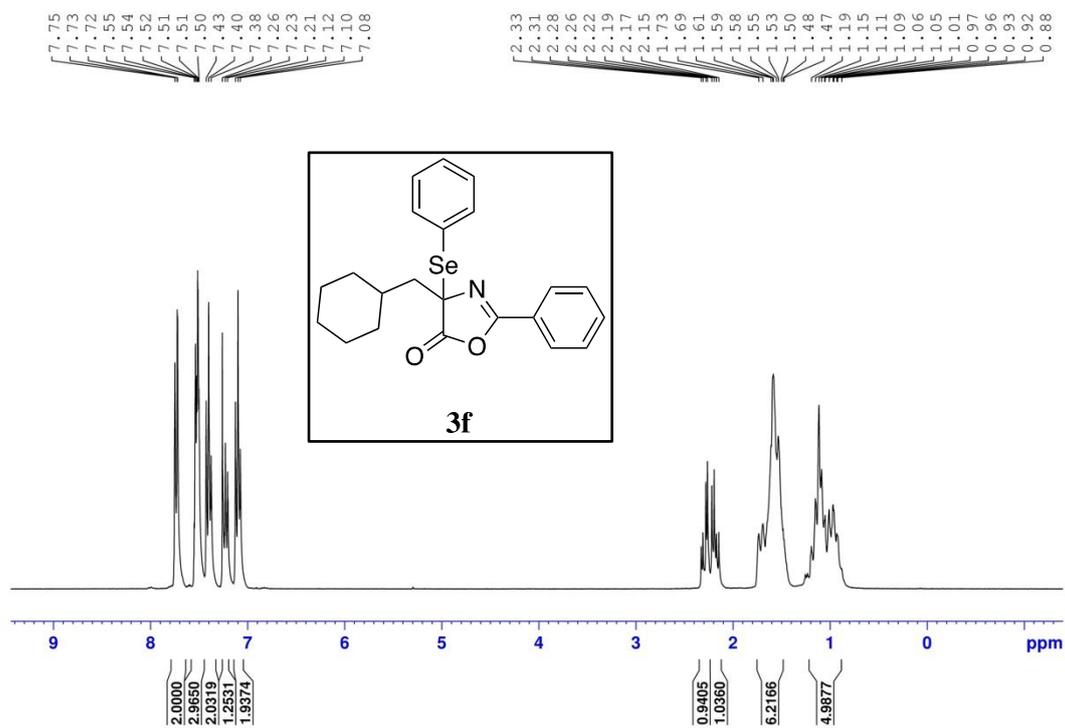
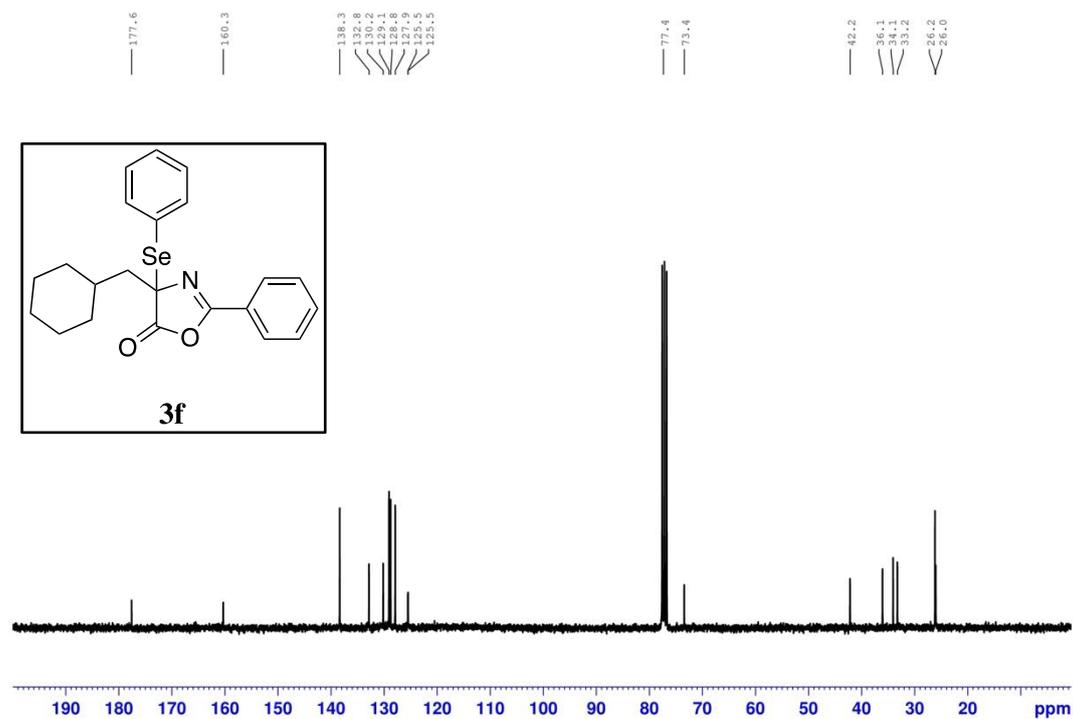
$^{13}\text{C-NMR}$ (75 MHz, CDCl_3 , 298 K)

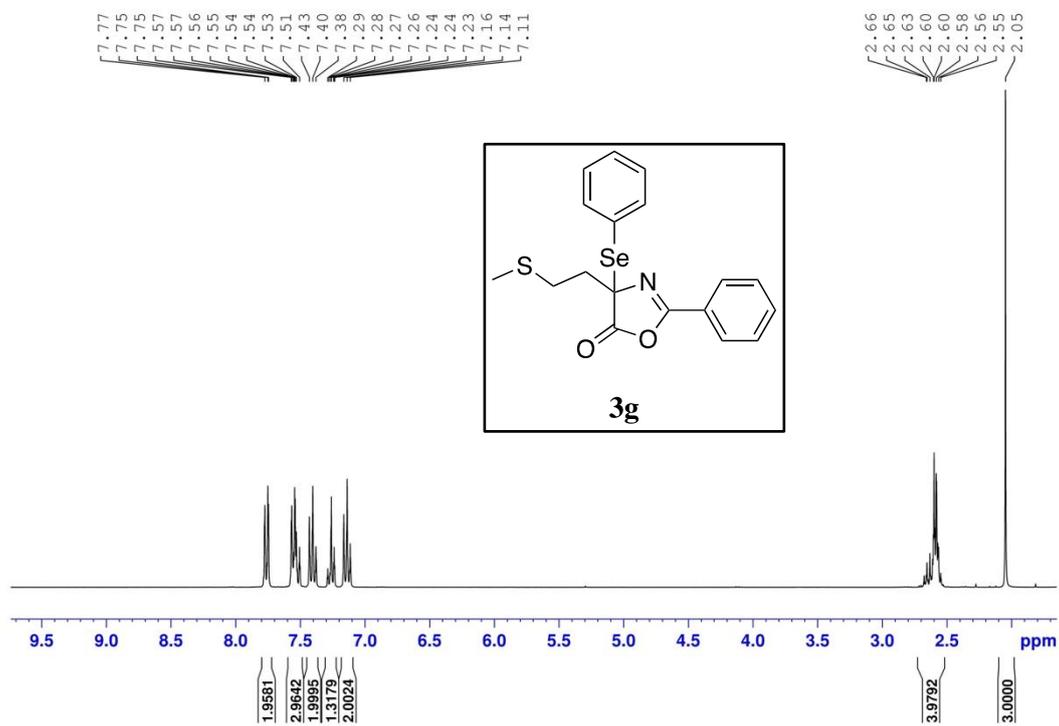
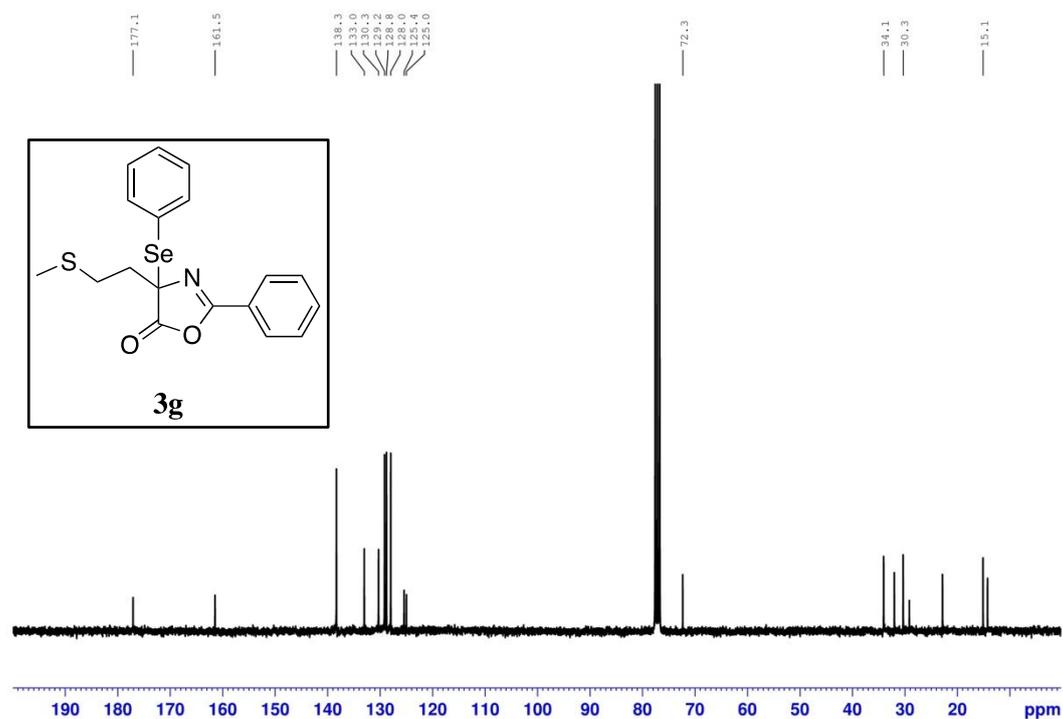


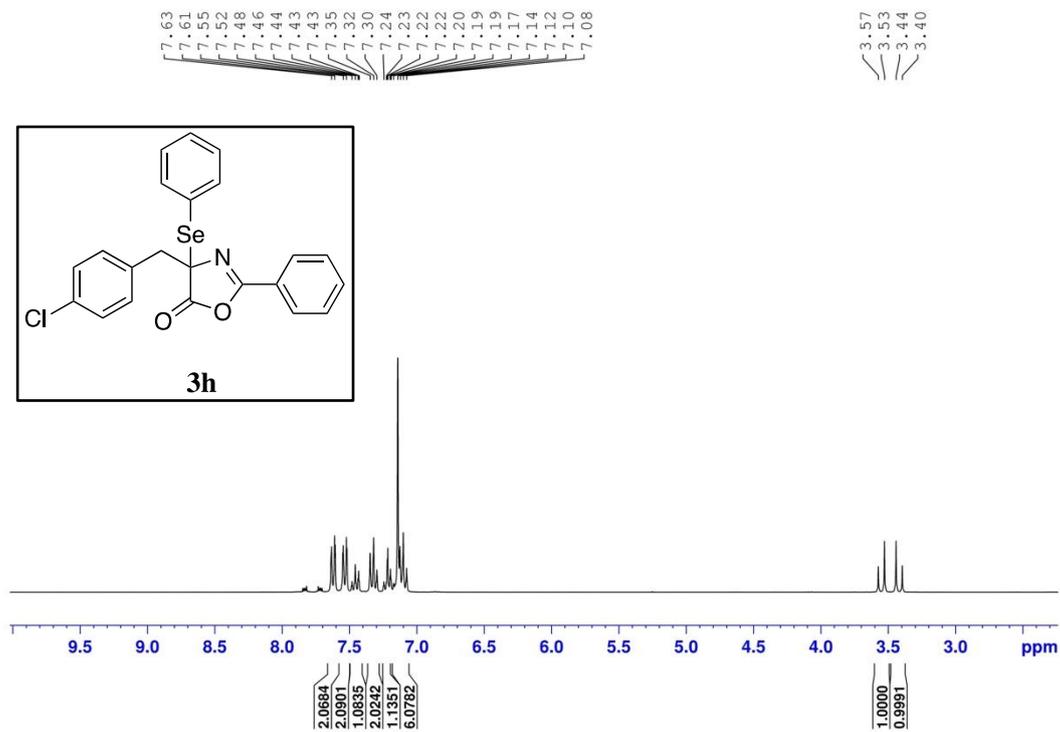
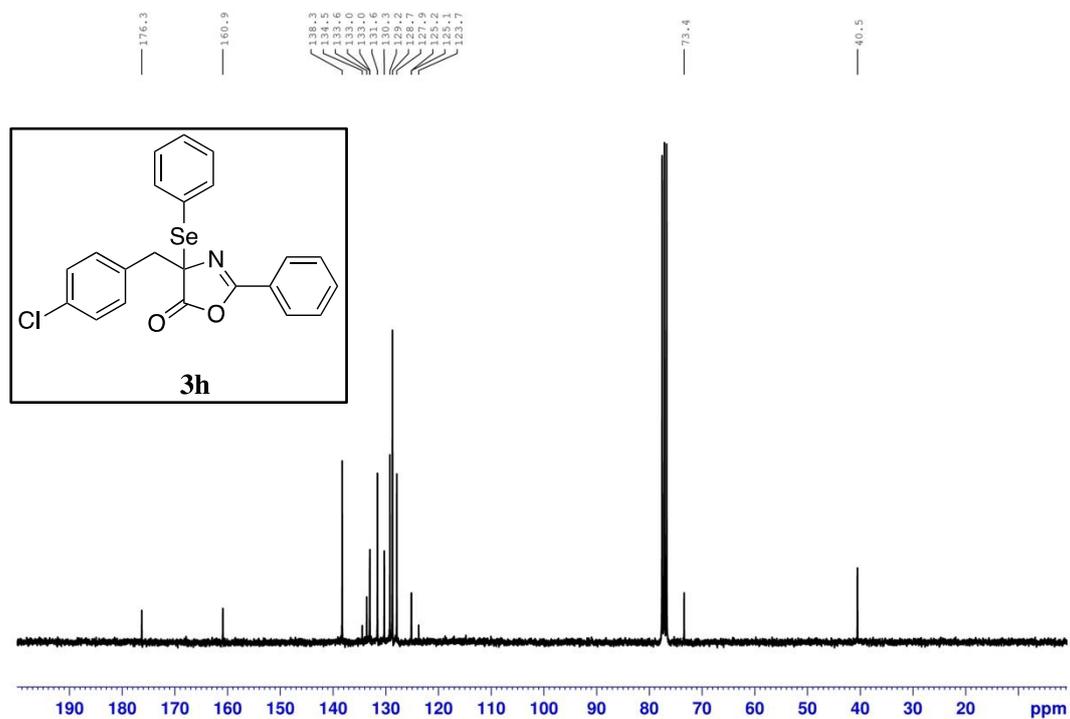
NMR spectra of compound 3c **$^1\text{H-NMR}$ (700 MHz, CDCl_3 , 298 K)** **$^{13}\text{C-NMR}$ (176 MHz, CDCl_3 , 298 K)**

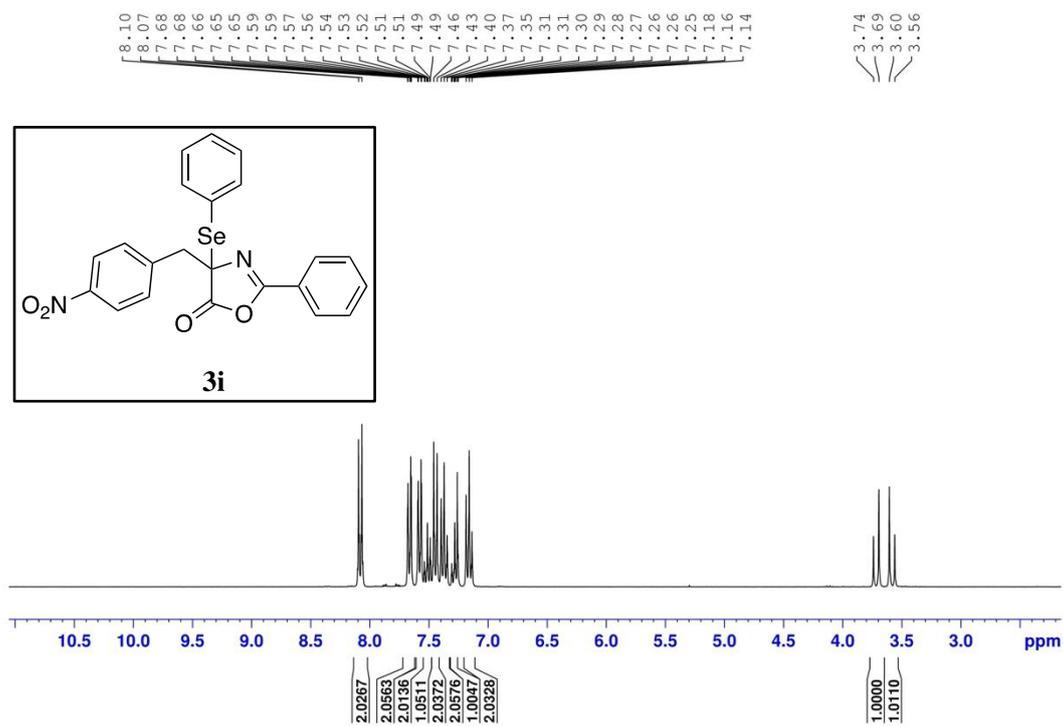
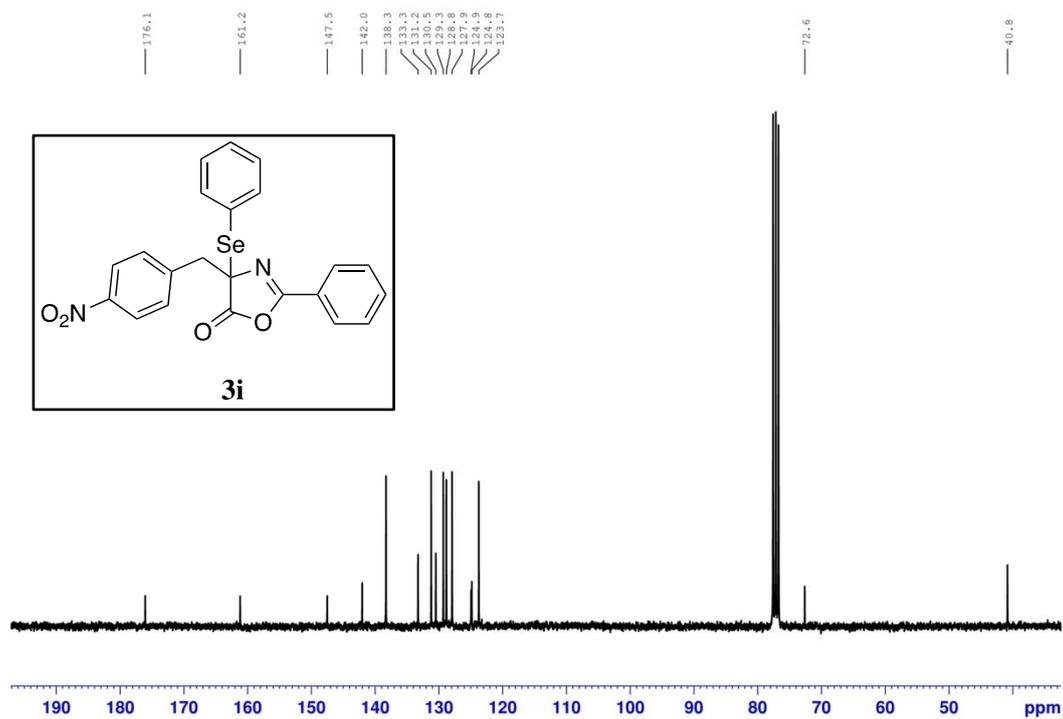
NMR spectra of compound 3d **$^1\text{H-NMR}$ (500 MHz, CDCl_3 , 298 K)** **$^{13}\text{C-NMR}$ (126 MHz, CDCl_3 , 298 K)**

NMR spectra of compound 3e **$^1\text{H-NMR}$** (500 MHz, CDCl_3 , 298 K) **$^{13}\text{C-NMR}$** (126 MHz, CDCl_3 , 298 K)

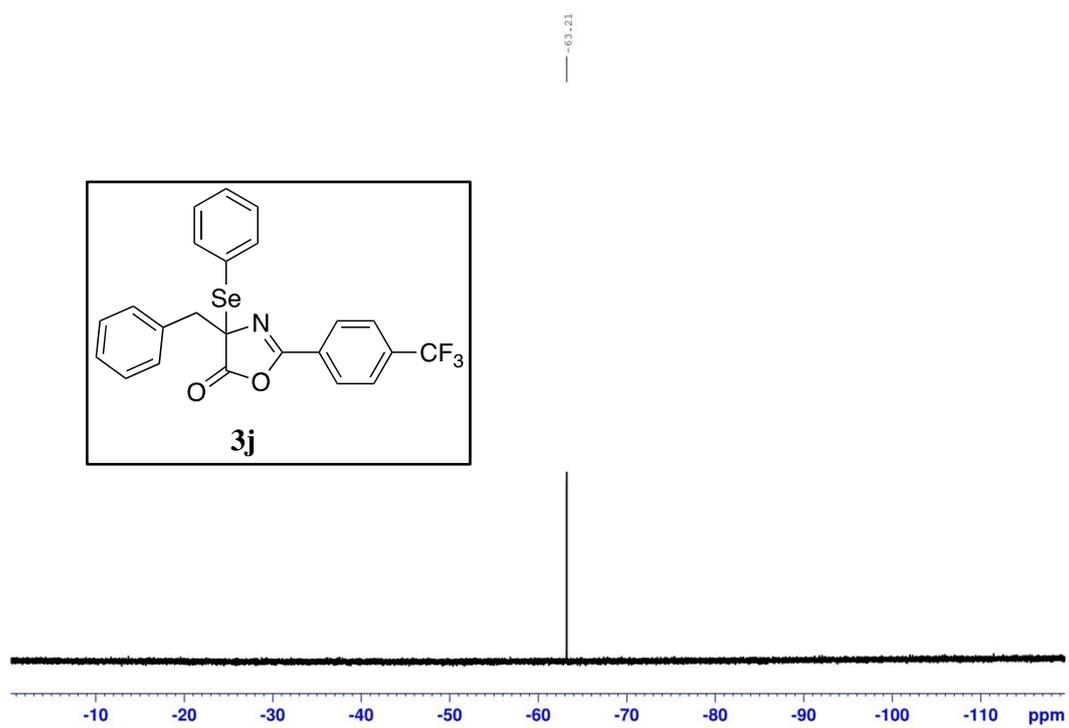
NMR spectra of compound 3f **$^1\text{H-NMR}$ (300 MHz, CDCl_3 , 298 K)** **$^{13}\text{C-NMR}$ (75 MHz, CDCl_3 , 298 K)**

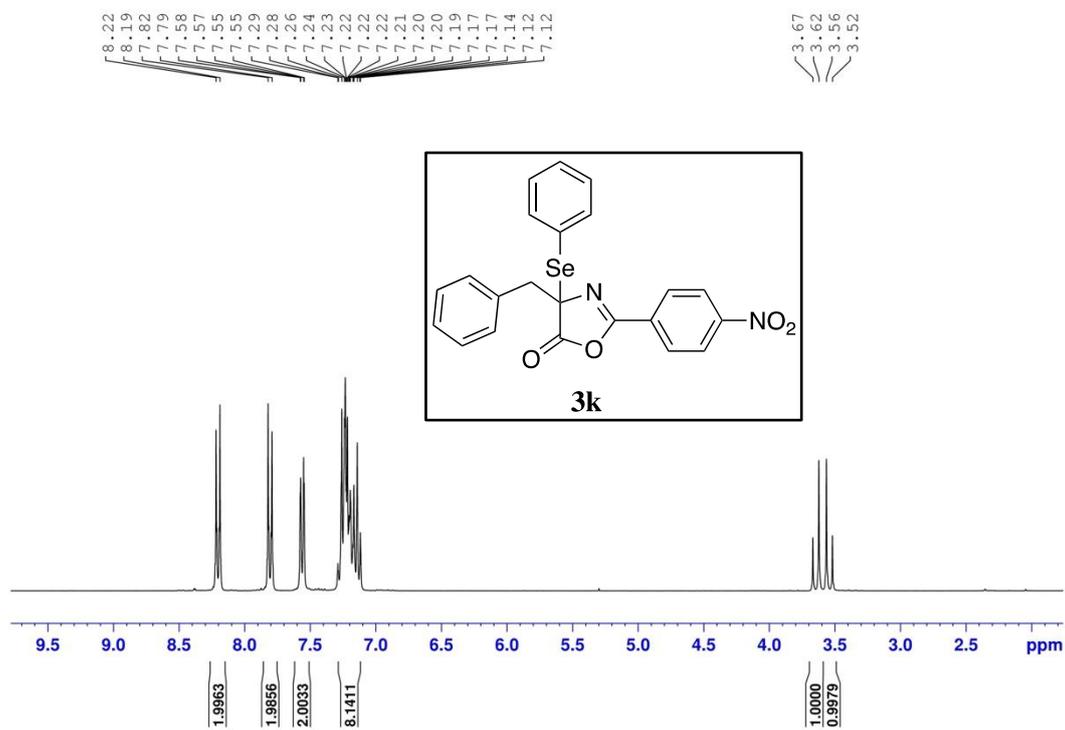
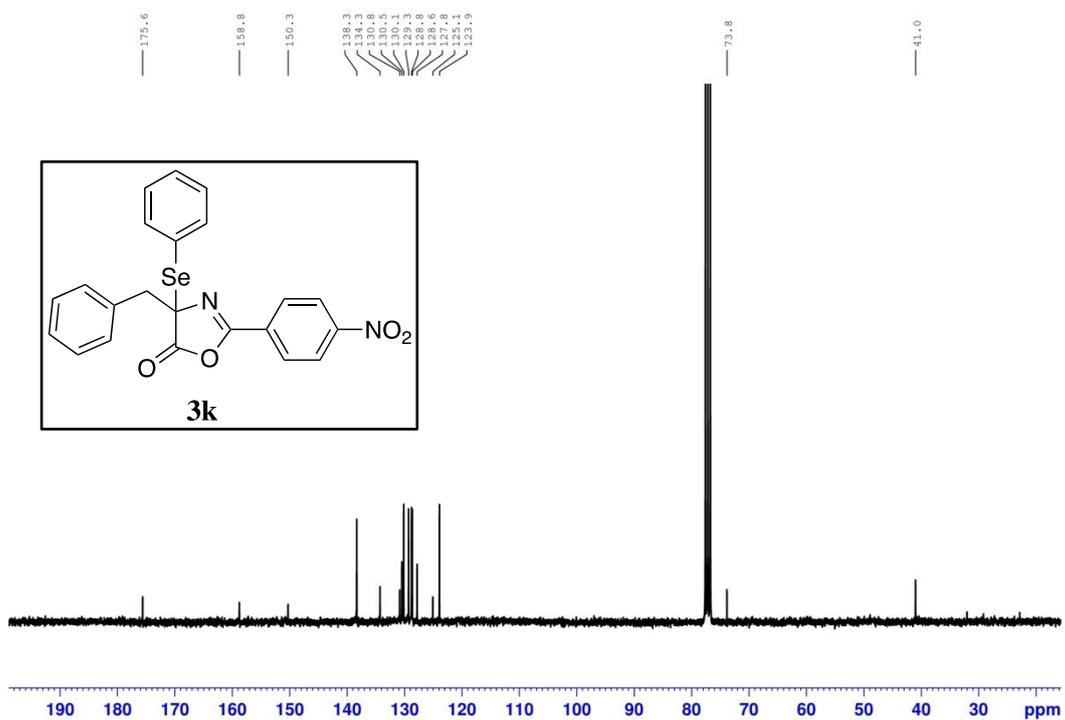
NMR spectra of compound 3g **$^1\text{H-NMR}$ (300 MHz, CDCl_3 , 298 K)** **$^{13}\text{C-NMR}$ (75 MHz, CDCl_3 , 298 K)**

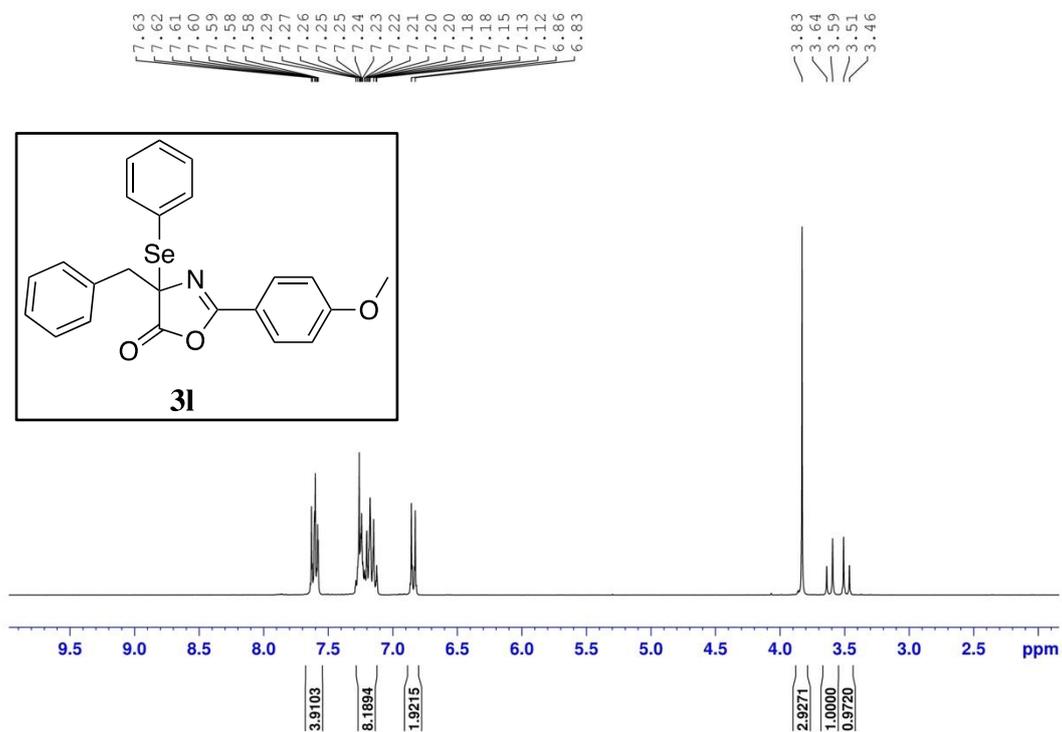
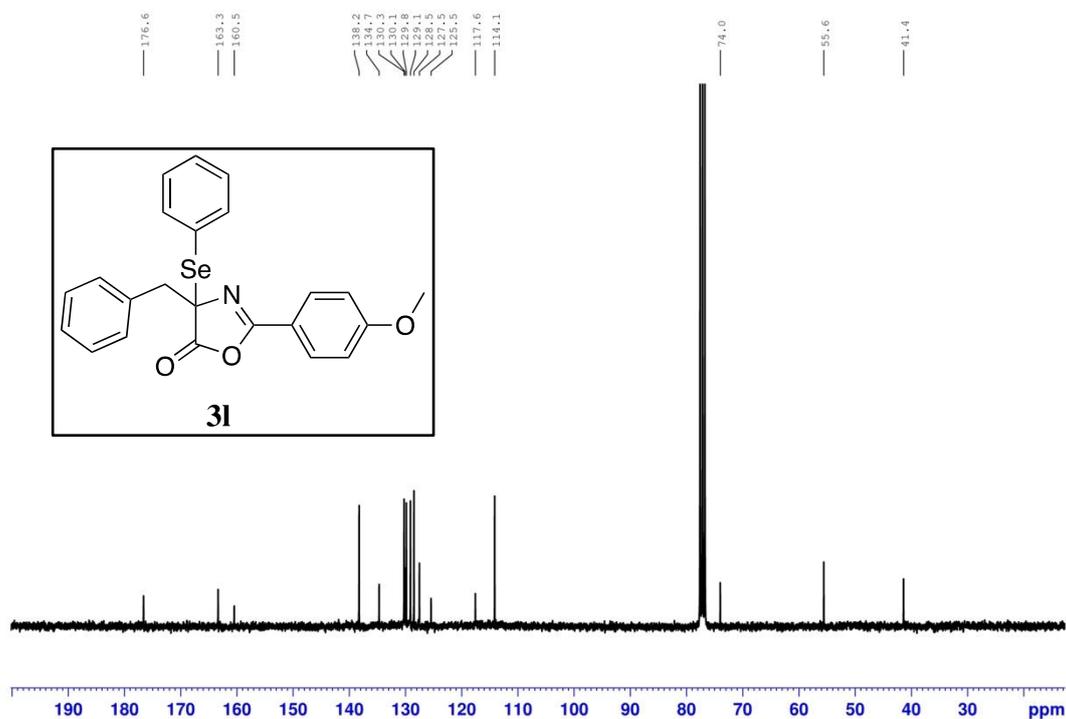
NMR spectra of compound 3h **$^1\text{H-NMR}$ (300 MHz, CDCl_3 , 298 K)** **$^{13}\text{C-NMR}$ (75 MHz, CDCl_3 , 298 K)**

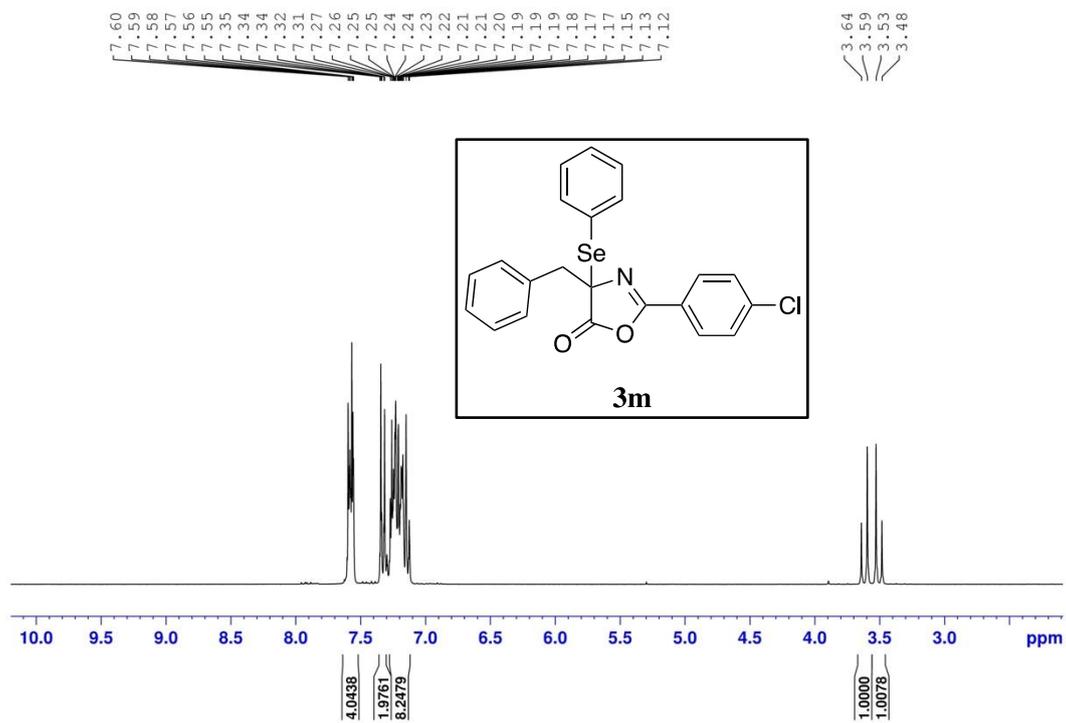
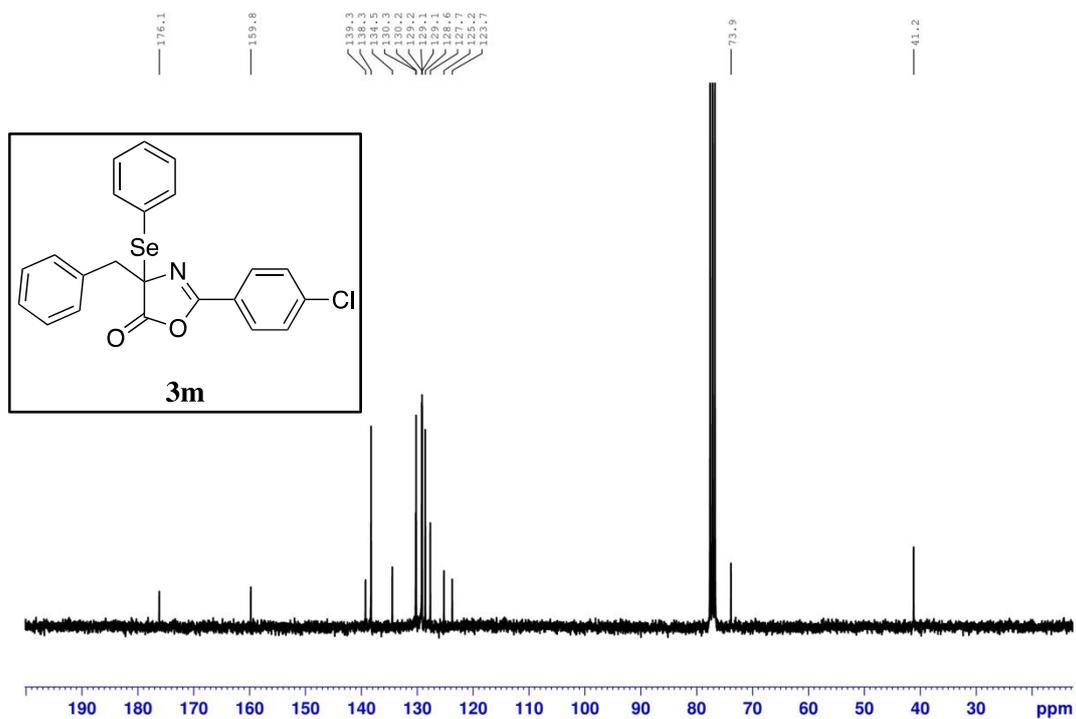
NMR spectra of compound 3i **$^1\text{H-NMR}$ (300 MHz, CDCl_3 , 298 K)** **$^{13}\text{C-NMR}$ (75 MHz, CDCl_3 , 298 K)**

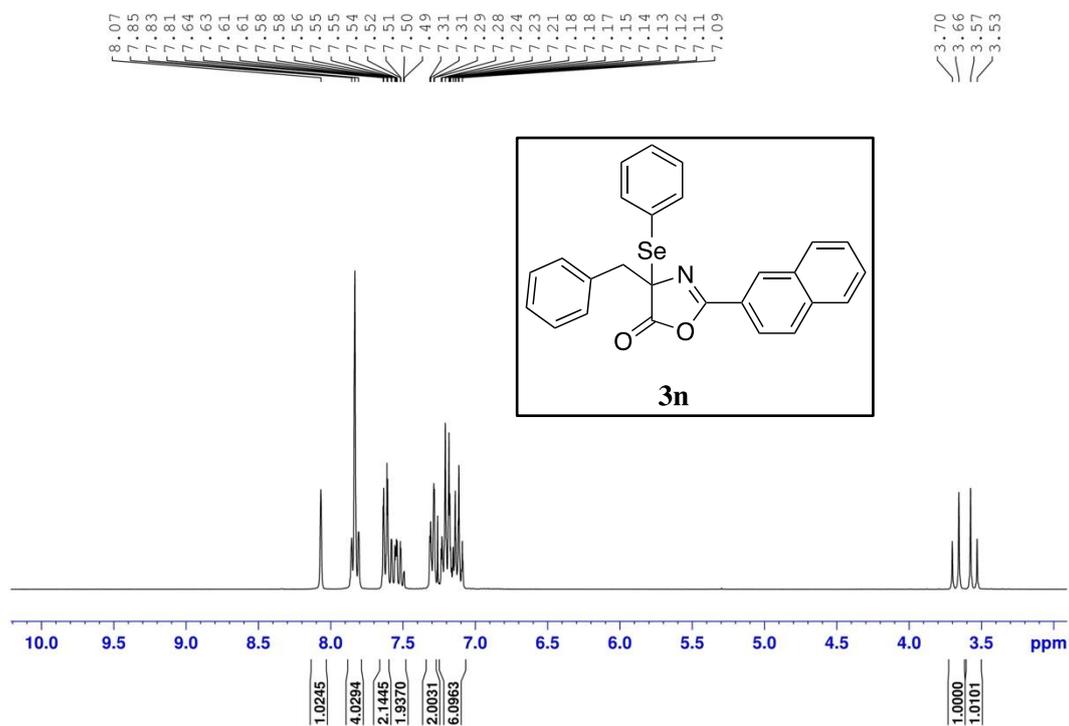
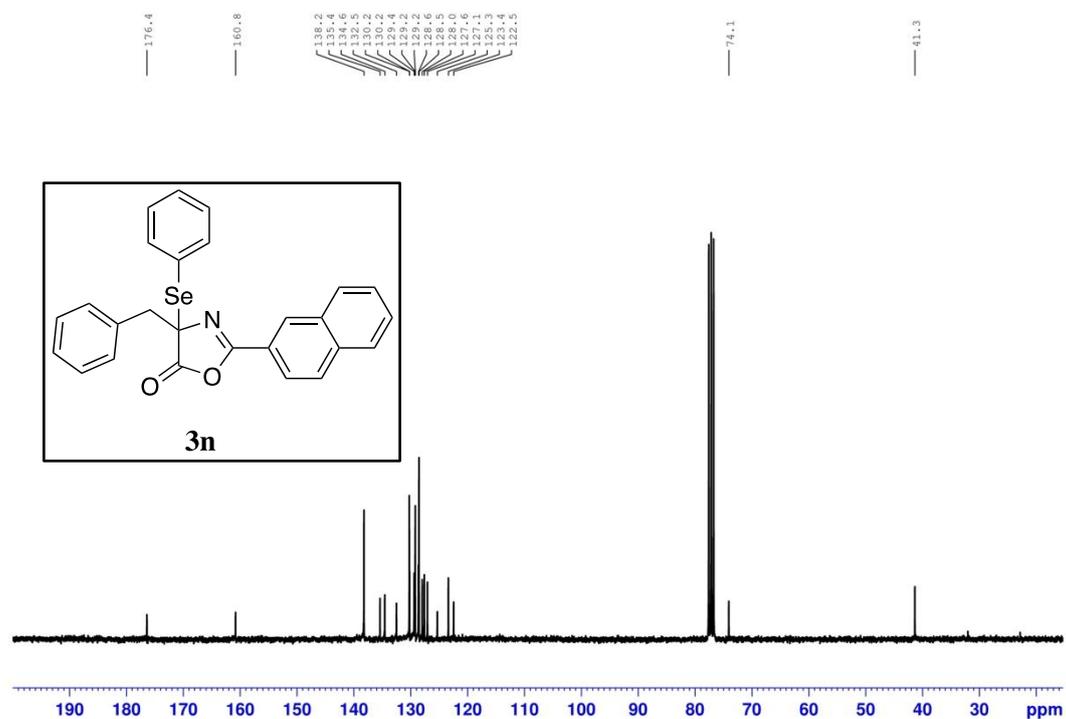
^{19}F -NMR (471 MHz, CDCl_3 , 298 K)

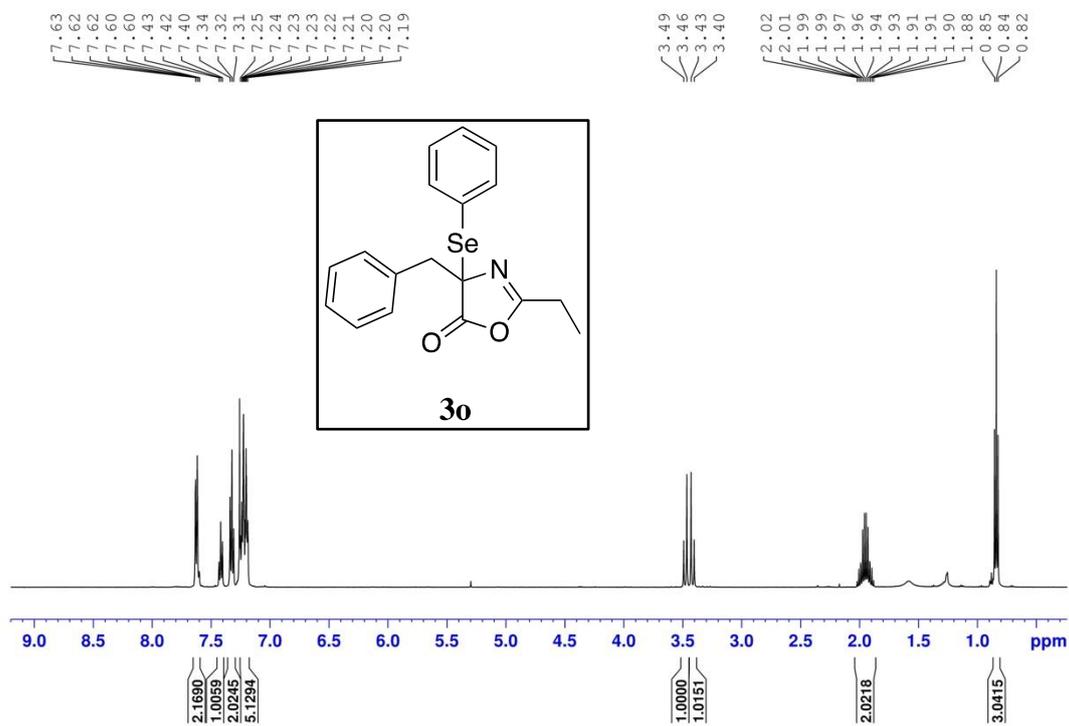
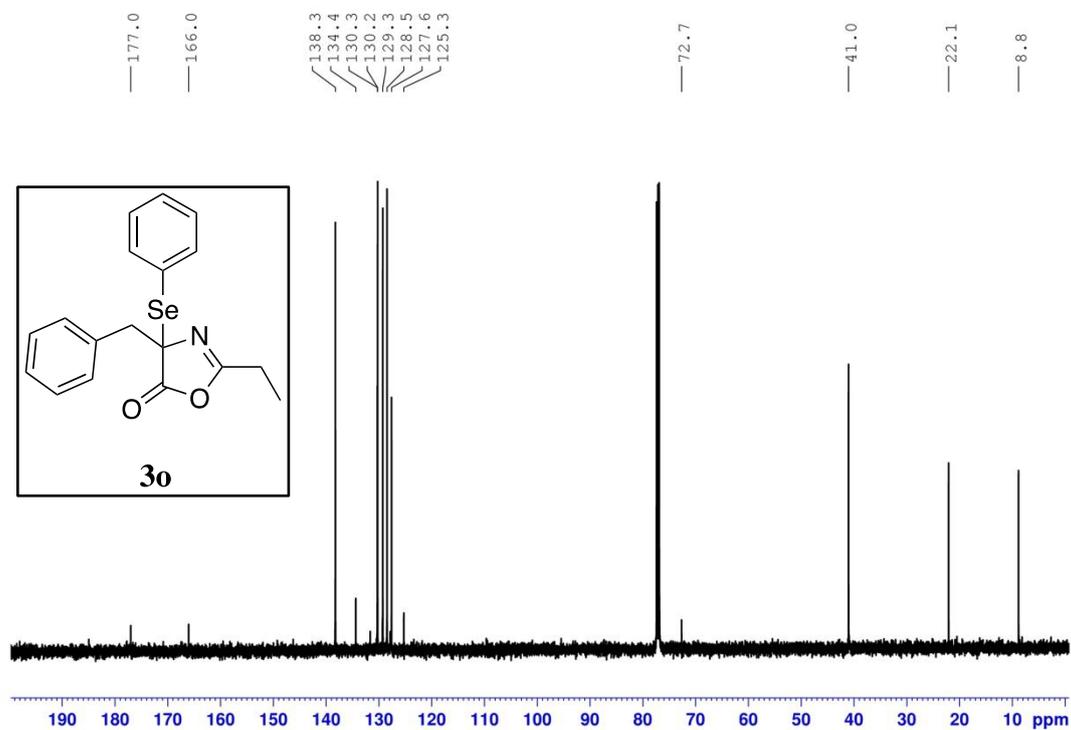


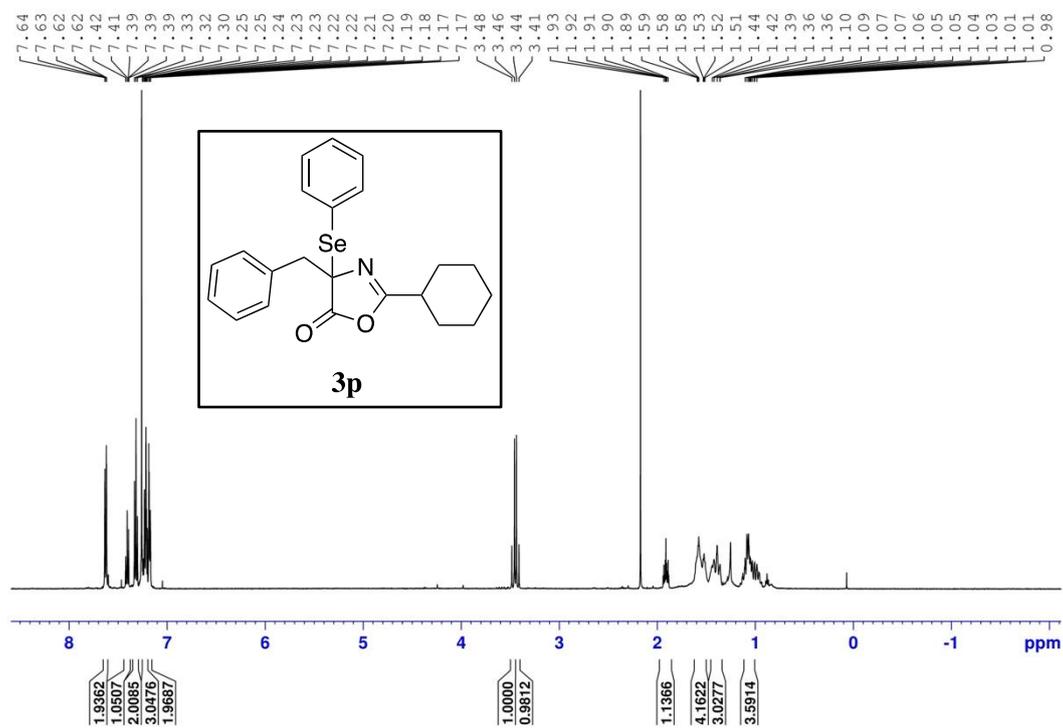
NMR spectra of compound 3k **$^1\text{H-NMR}$ (300 MHz, CDCl_3 , 298 K)** **$^{13}\text{C-NMR}$ (75 MHz, CDCl_3 , 298 K)**

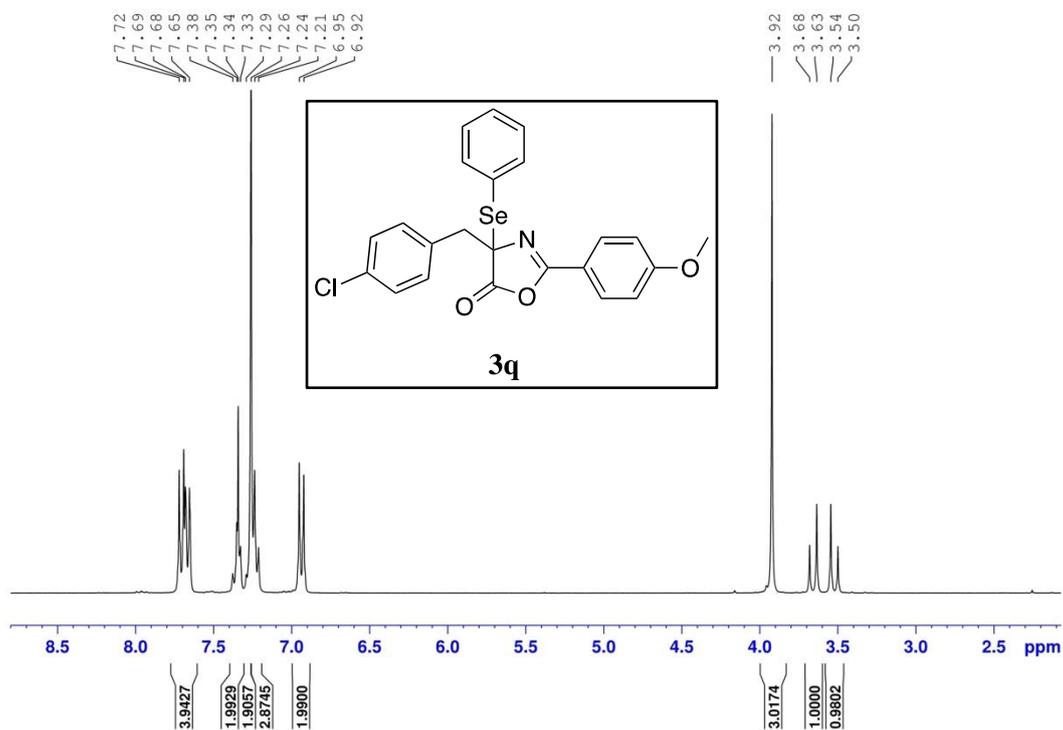
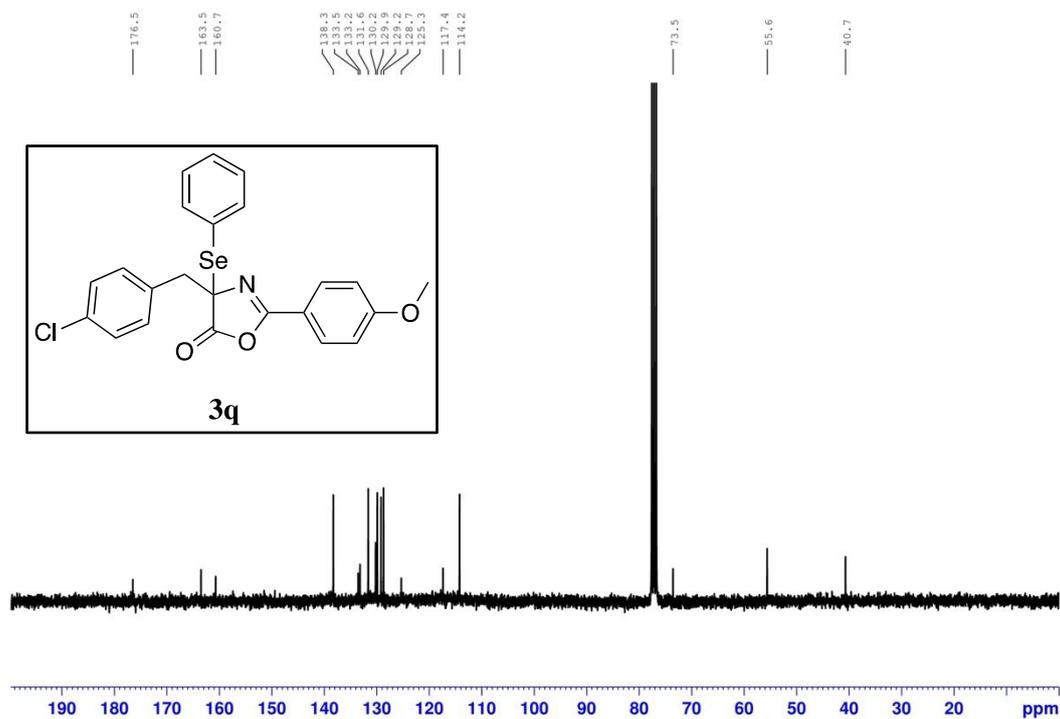
NMR spectra of compound 31 **$^1\text{H-NMR}$ (300 MHz, CDCl_3 , 298 K)** **$^{13}\text{C-NMR}$ (75 MHz, CDCl_3 , 298 K)**

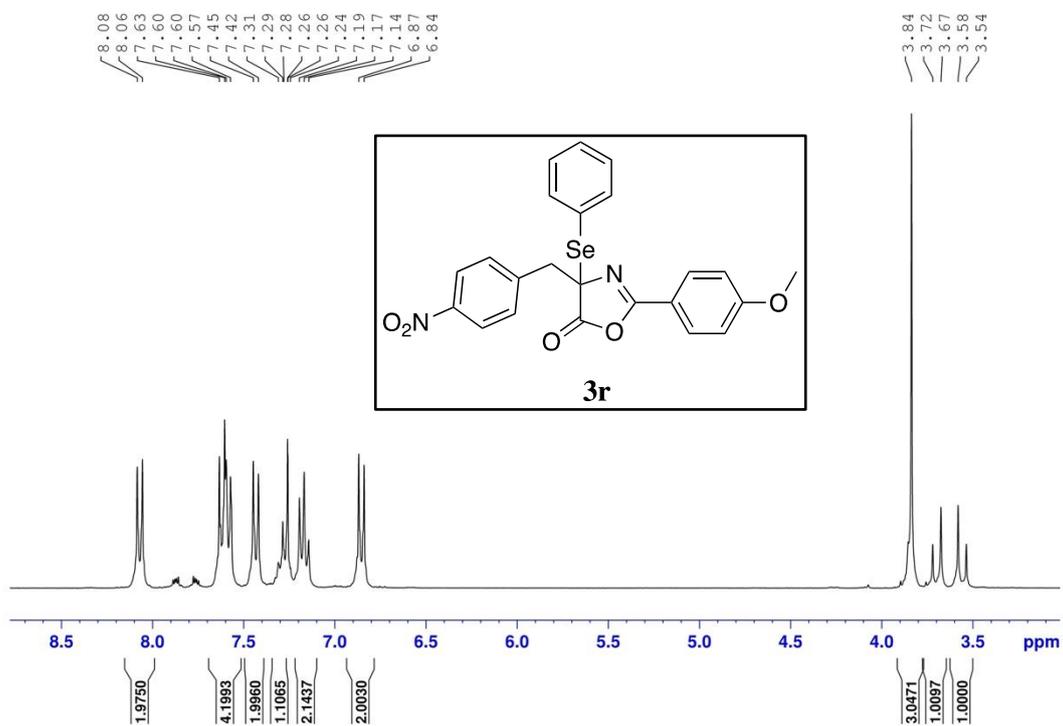
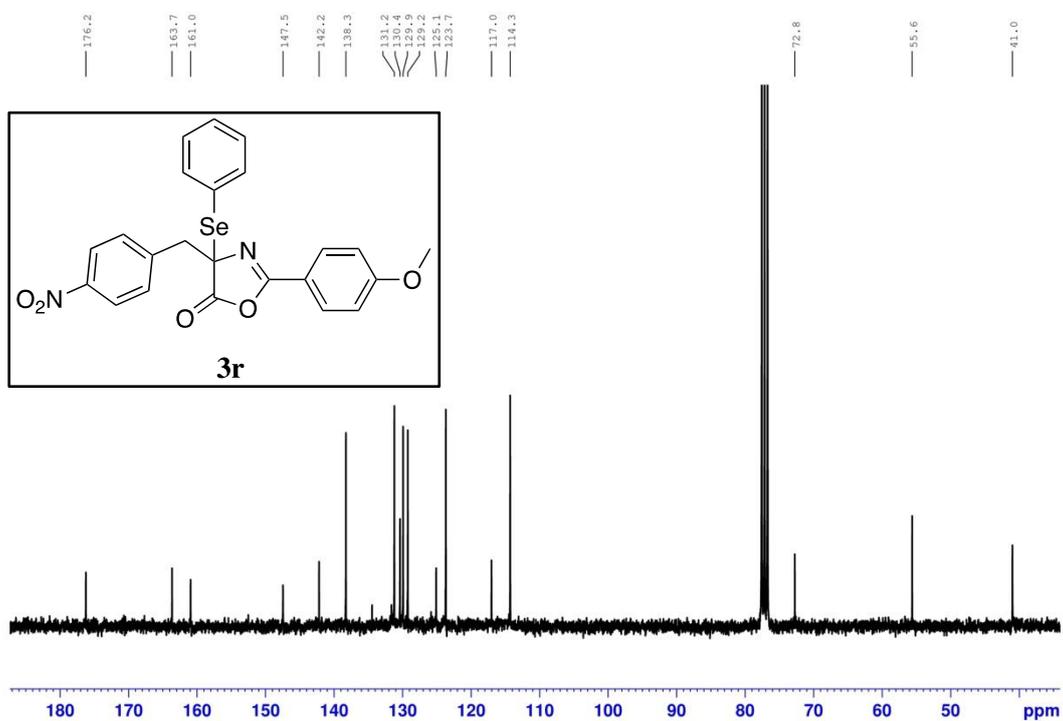
NMR spectra of compound 3m **$^1\text{H-NMR}$ (300 MHz, CDCl_3 , 298 K)** **$^{13}\text{C-NMR}$ (75 MHz, CDCl_3 , 298 K)**

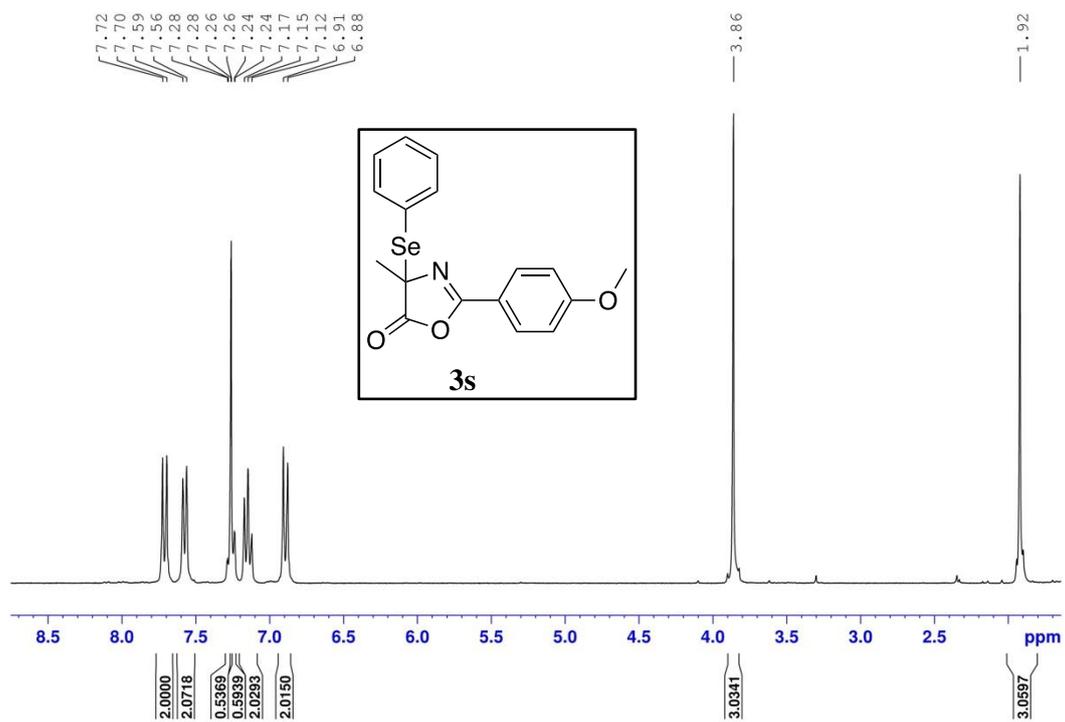
NMR spectra of compound 3n **$^1\text{H-NMR}$ (300 MHz, CDCl_3 , 298 K)** **$^{13}\text{C-NMR}$ (75 MHz, CDCl_3 , 298 K)**

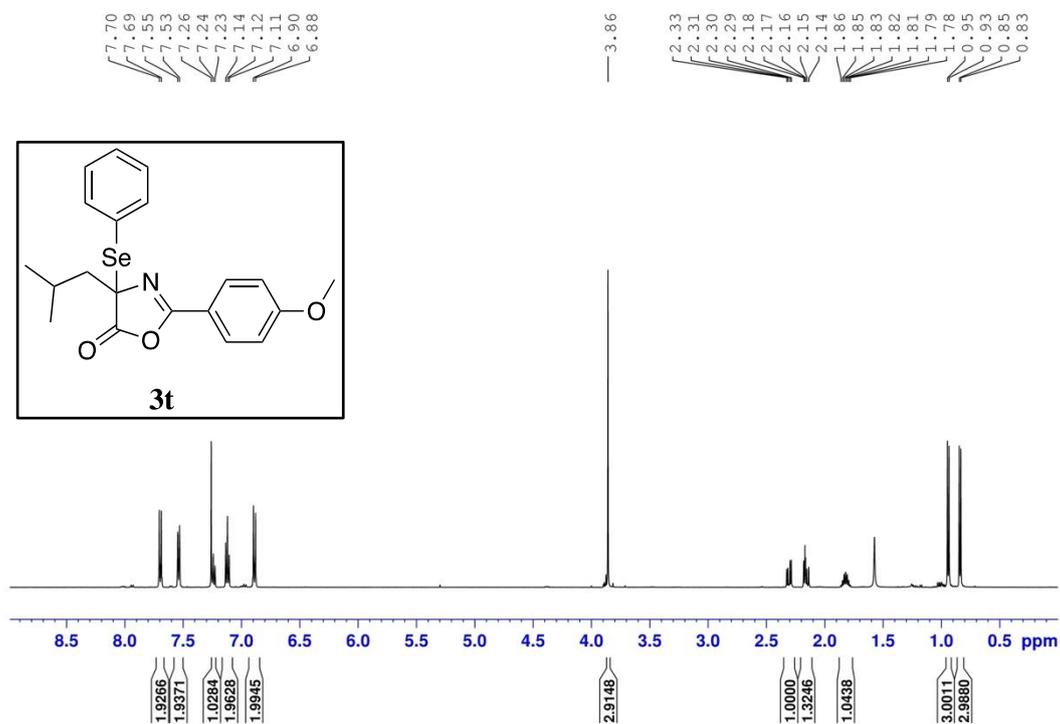
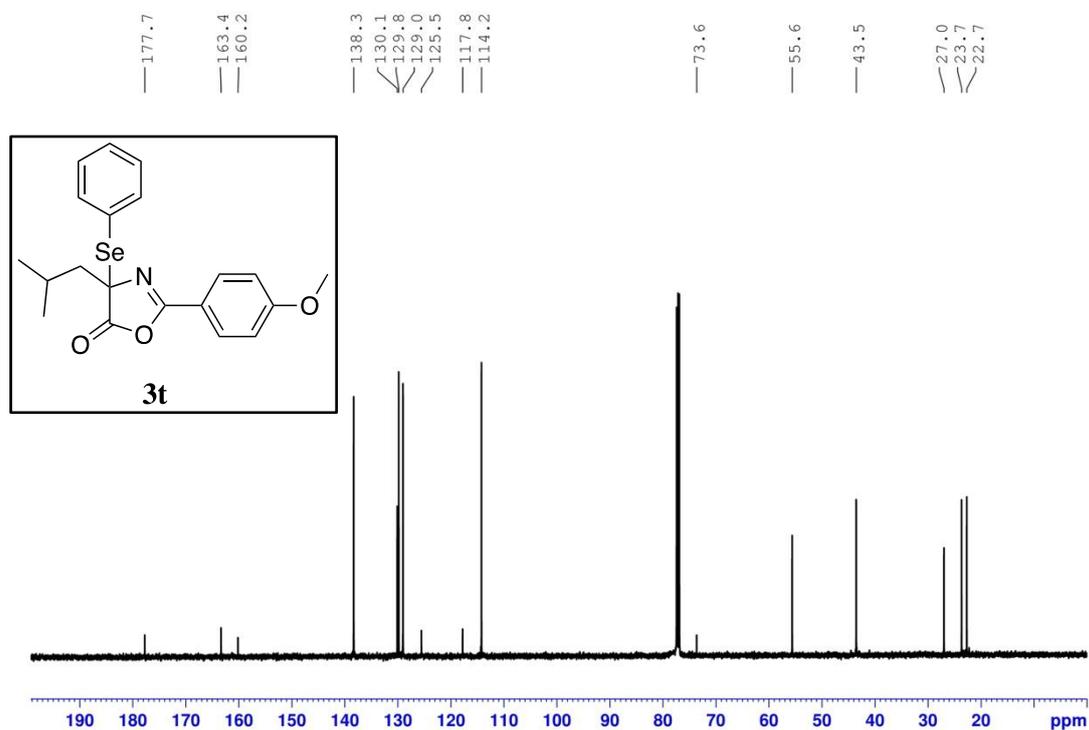
NMR spectra of compound 3o **$^1\text{H-NMR}$ (500 MHz, CDCl_3 , 298 K)** **$^{13}\text{C-NMR}$ (126 MHz, CDCl_3 , 298 K)**

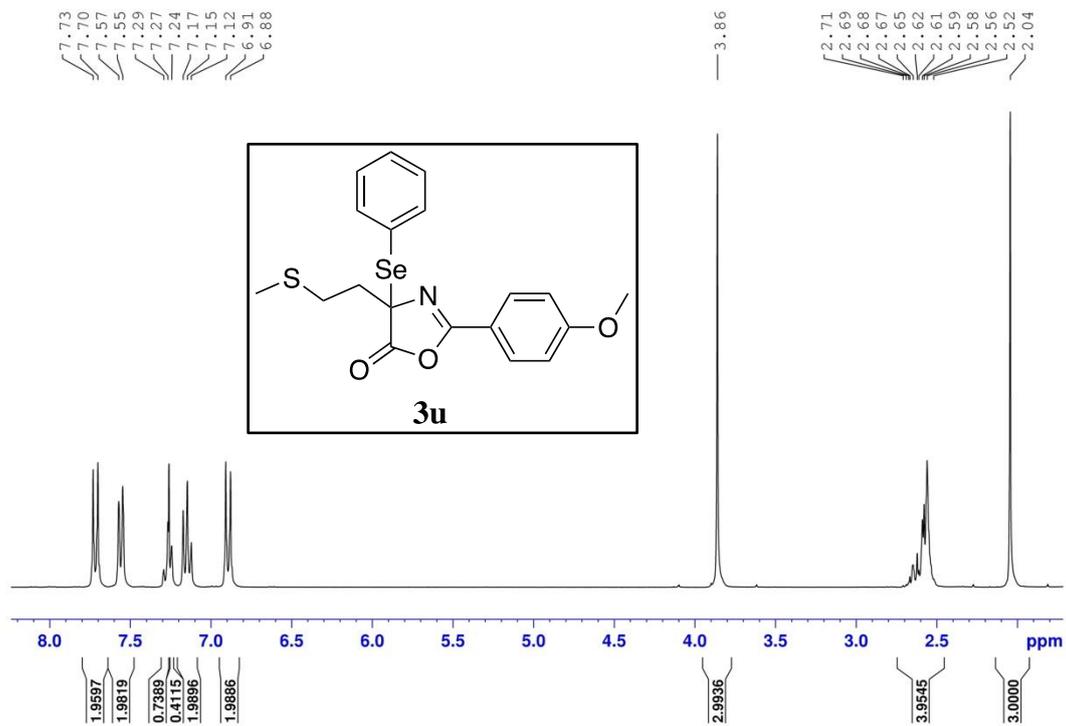
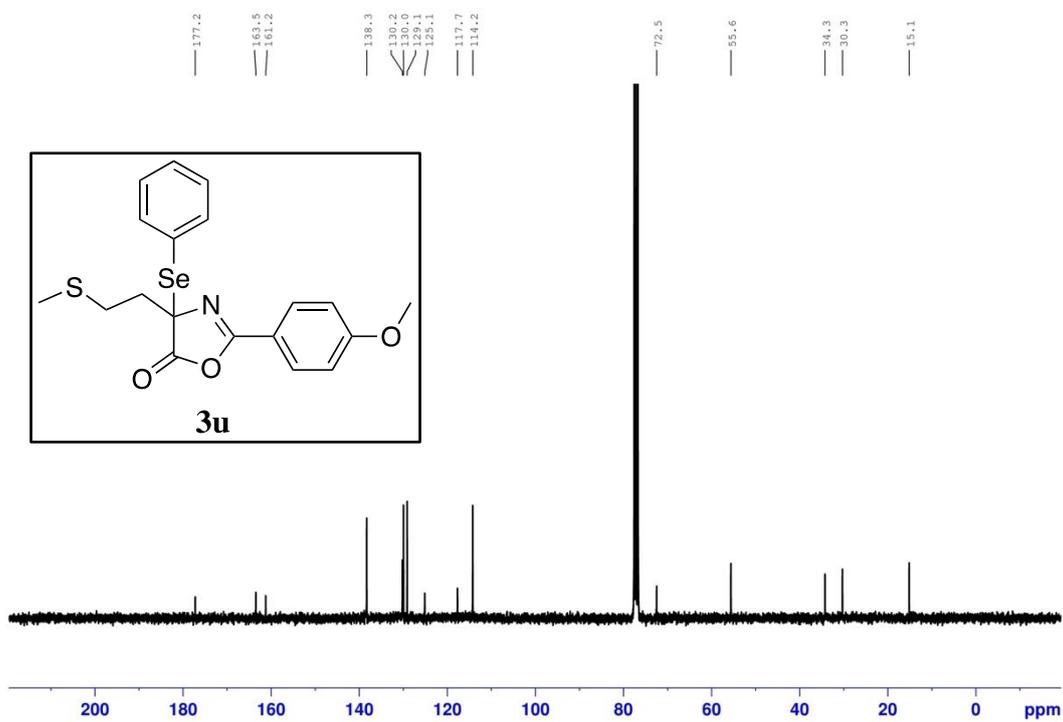
NMR spectra of compound 3p **$^1\text{H-NMR}$ (500 MHz, CDCl_3 , 298 K)**

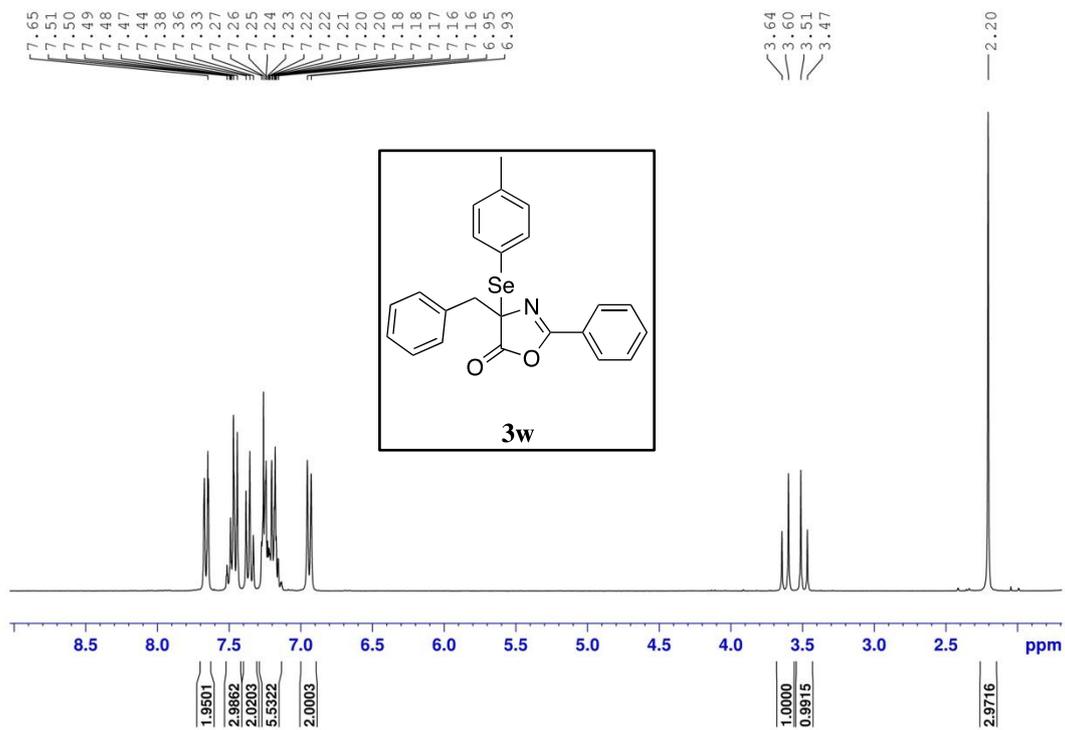
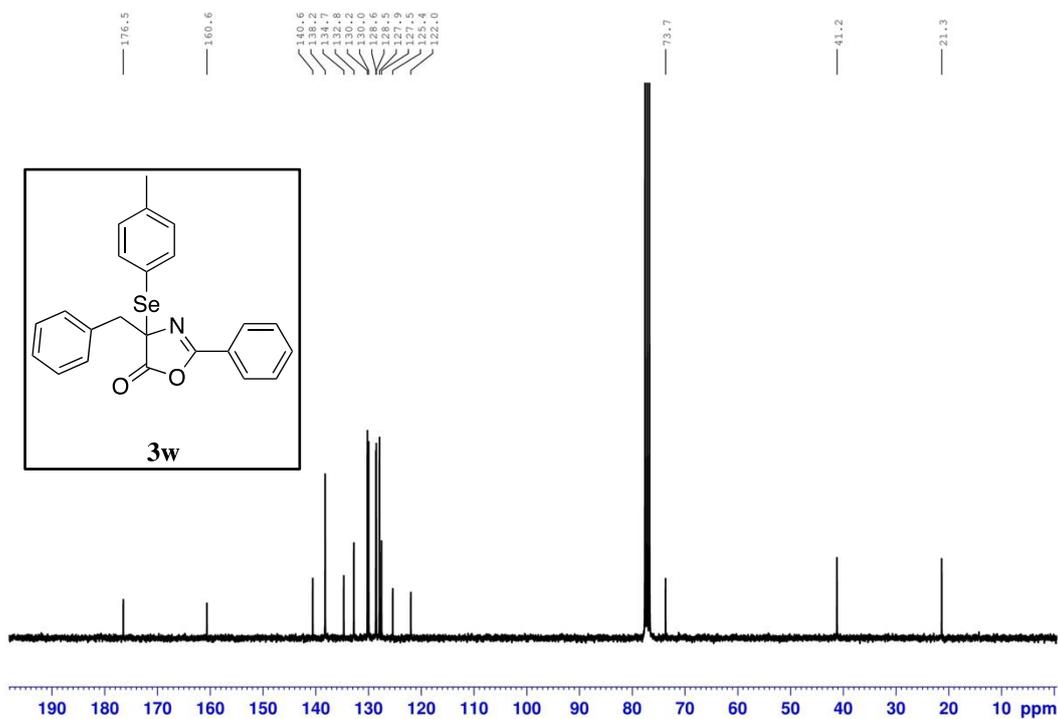
NMR spectra of compound 3q **$^1\text{H-NMR}$ (300 MHz, CDCl_3 , 298 K)** **$^{13}\text{C-NMR}$ (75 MHz, CDCl_3 , 298 K)**

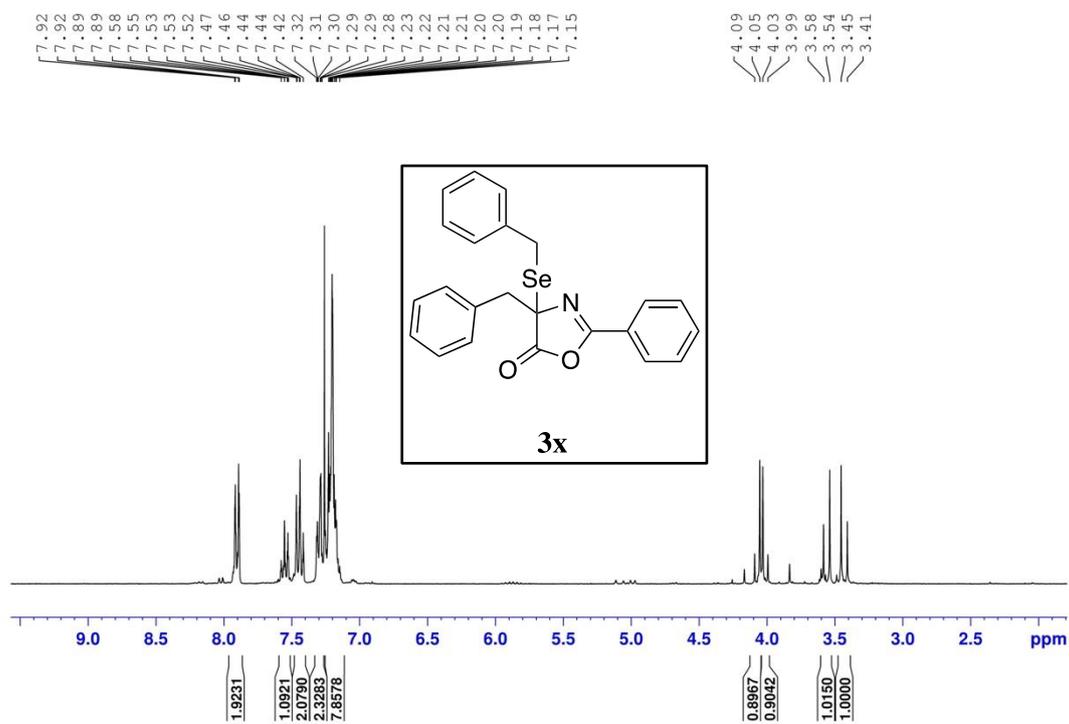
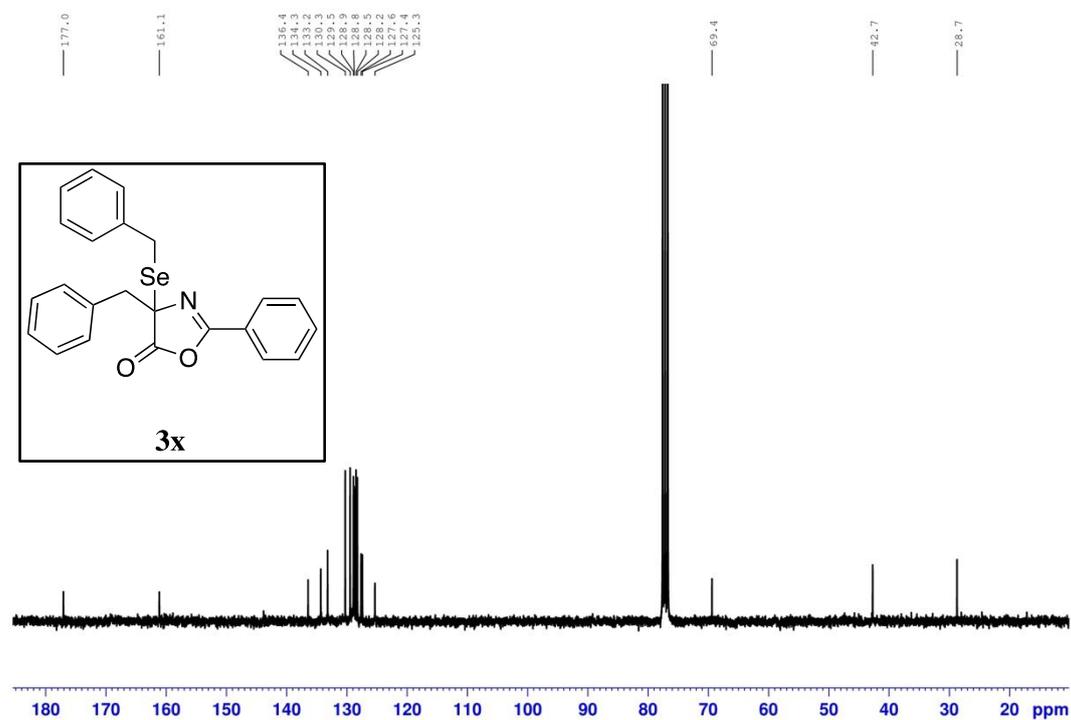
NMR spectra of compound 3r **$^1\text{H-NMR}$ (300 MHz, CDCl_3 , 298 K)** **$^{13}\text{C-NMR}$ (75 MHz, CDCl_3 , 298 K)**

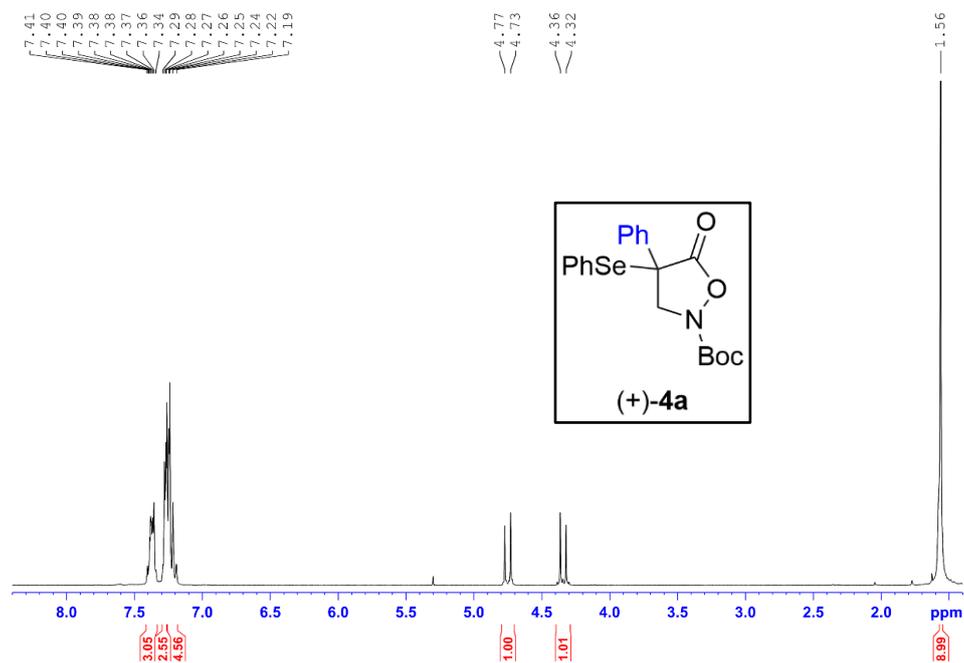
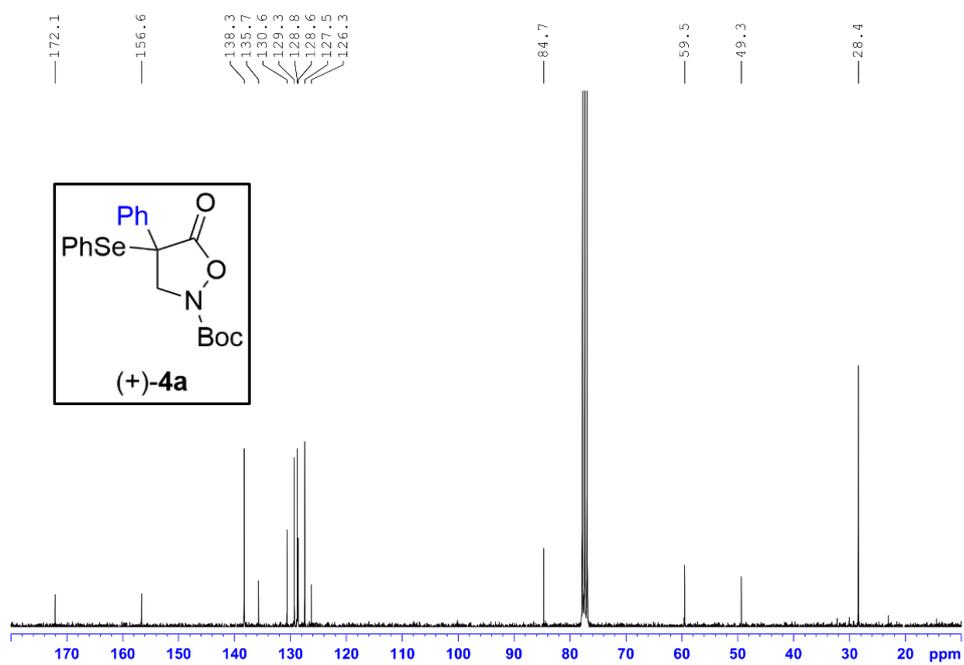
NMR spectra of compound 3s **$^1\text{H-NMR}$ (300 MHz, CDCl_3 , 298 K)**

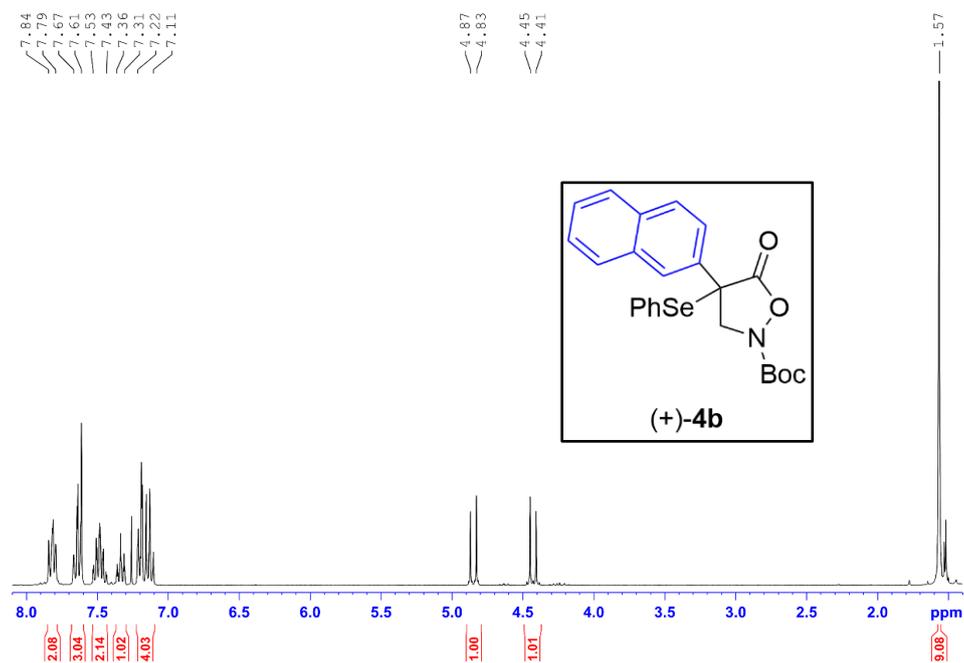
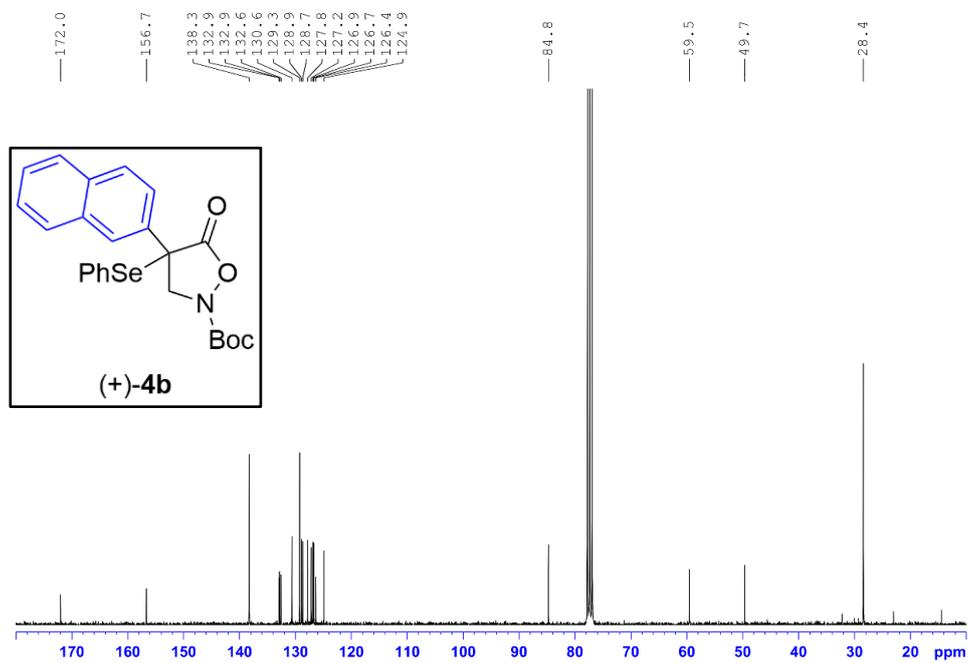
NMR spectra of compound 3t **$^1\text{H-NMR}$ (300 MHz, CDCl_3 , 298 K)** **$^{13}\text{C-NMR}$ (126 MHz, CDCl_3 , 298 K)**

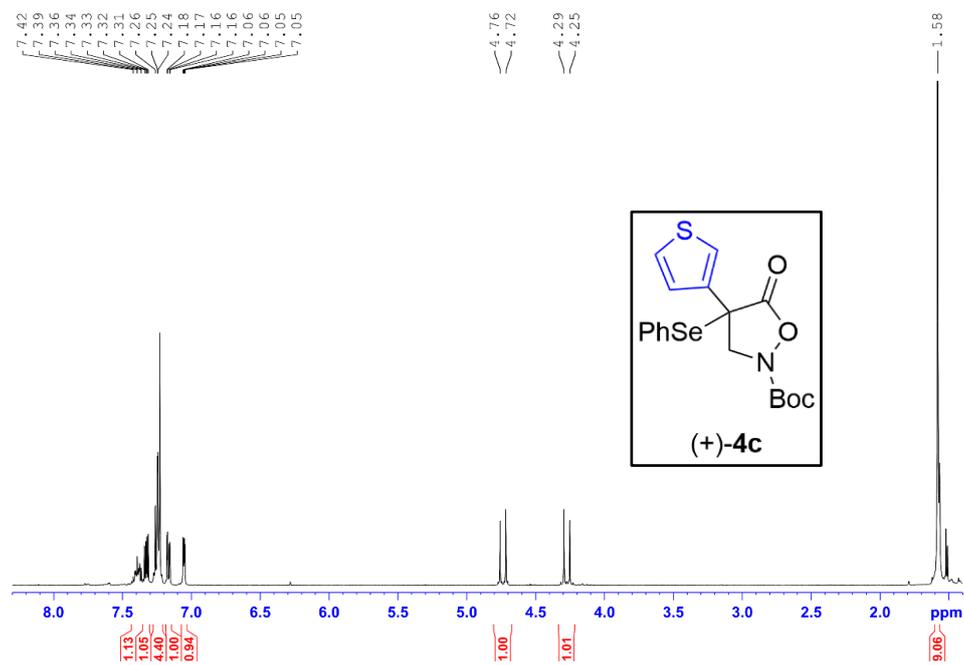
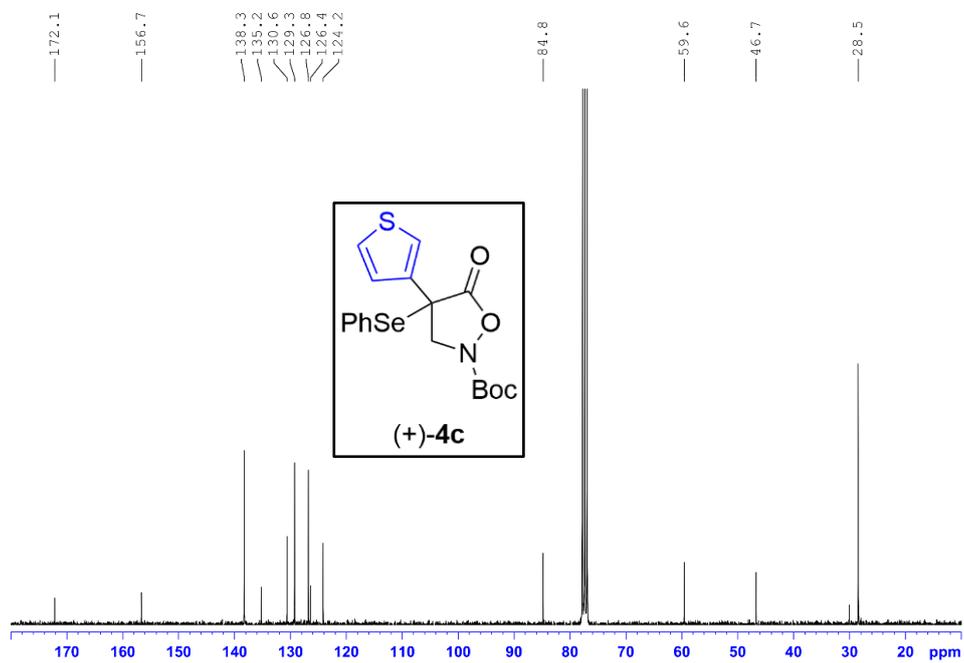
NMR spectra of compound 3u **$^1\text{H-NMR}$ (300 MHz, CDCl_3 , 298 K)** **$^{13}\text{C-NMR}$ (75 MHz, CDCl_3 , 298 K)**

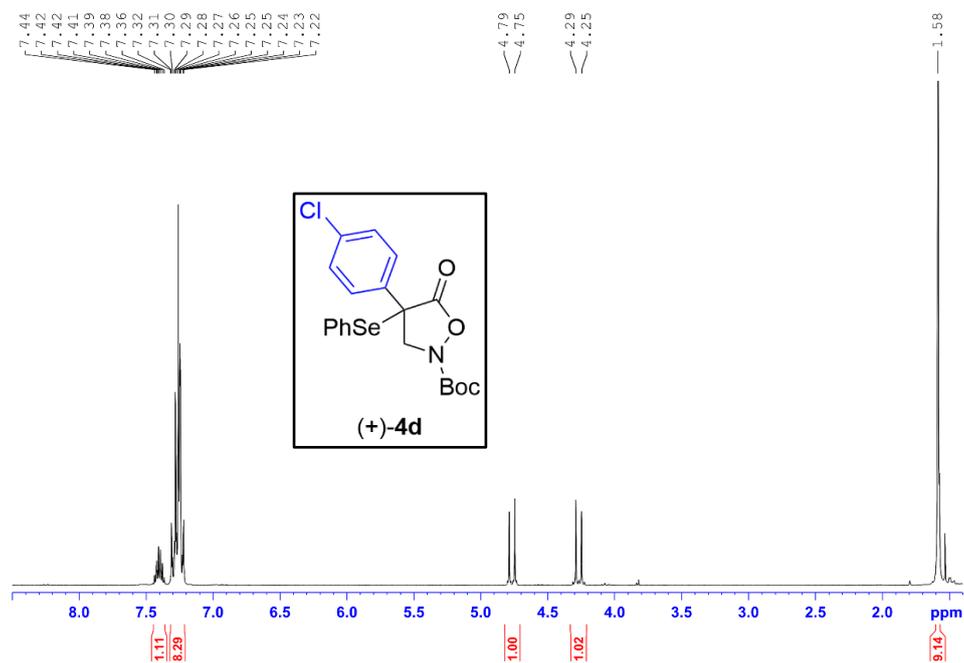
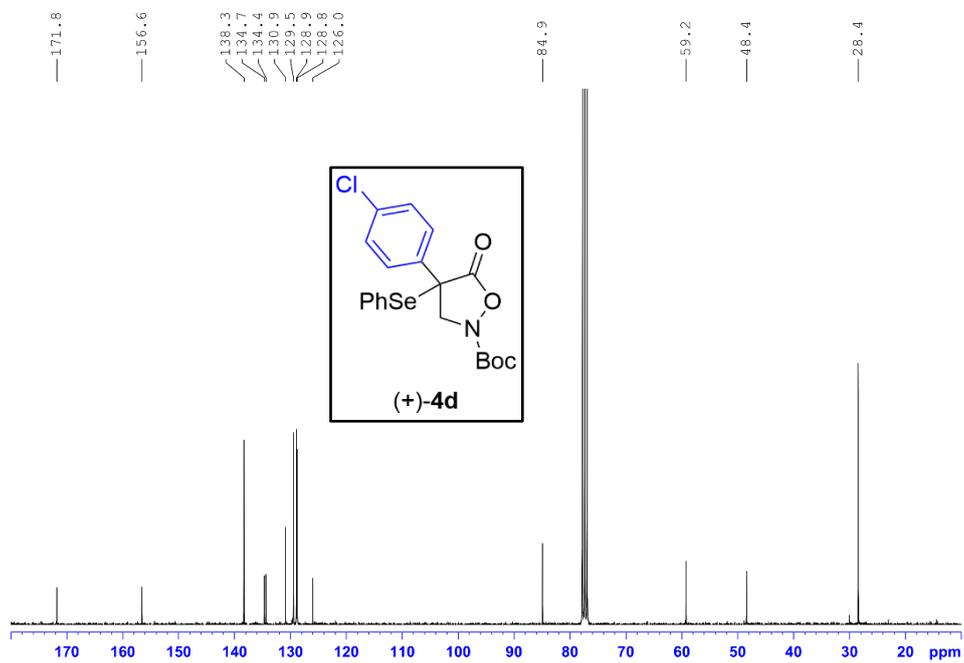
NMR spectra of compound 3w **$^1\text{H-NMR}$ (300 MHz, CDCl_3 , 298 K)** **$^{13}\text{C-NMR}$ (75 MHz, CDCl_3 , 298 K)**

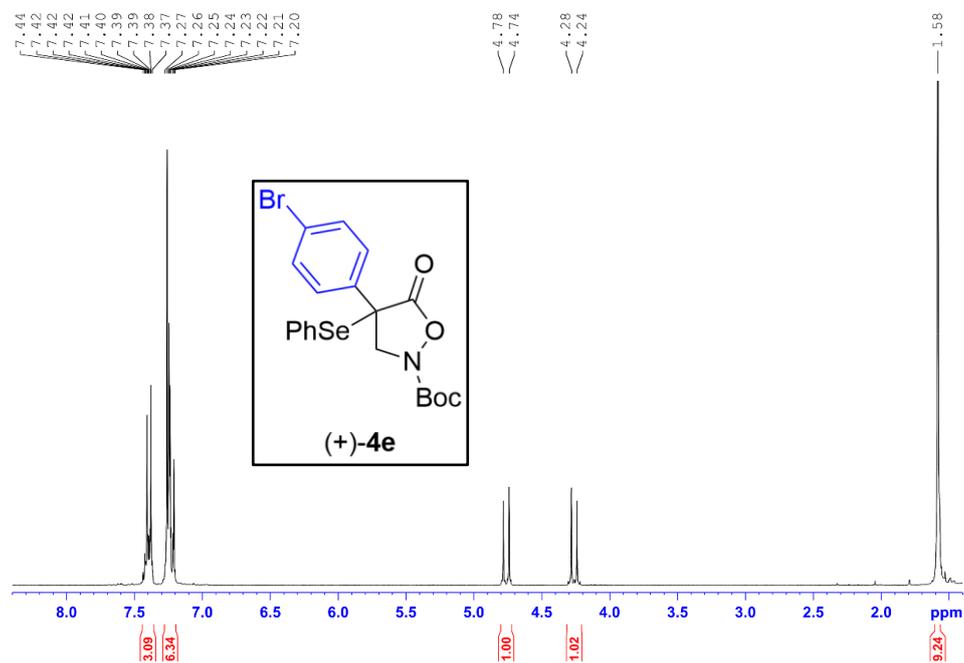
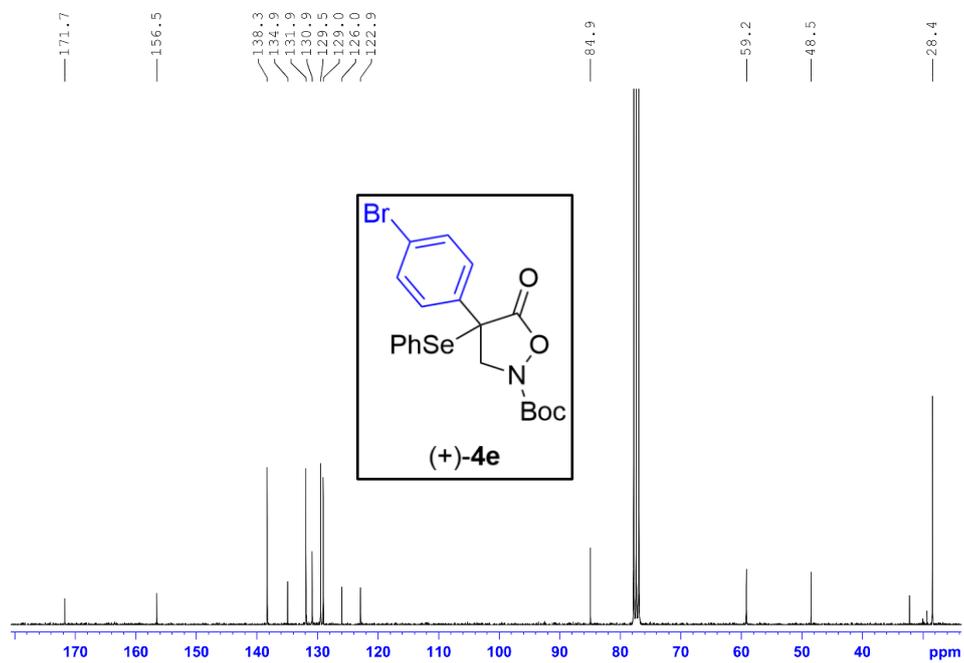
NMR spectra of compound 3x **$^1\text{H-NMR}$ (300 MHz, CDCl_3 , 298 K)** **$^{13}\text{C-NMR}$ (75 MHz, CDCl_3 , 298 K)**

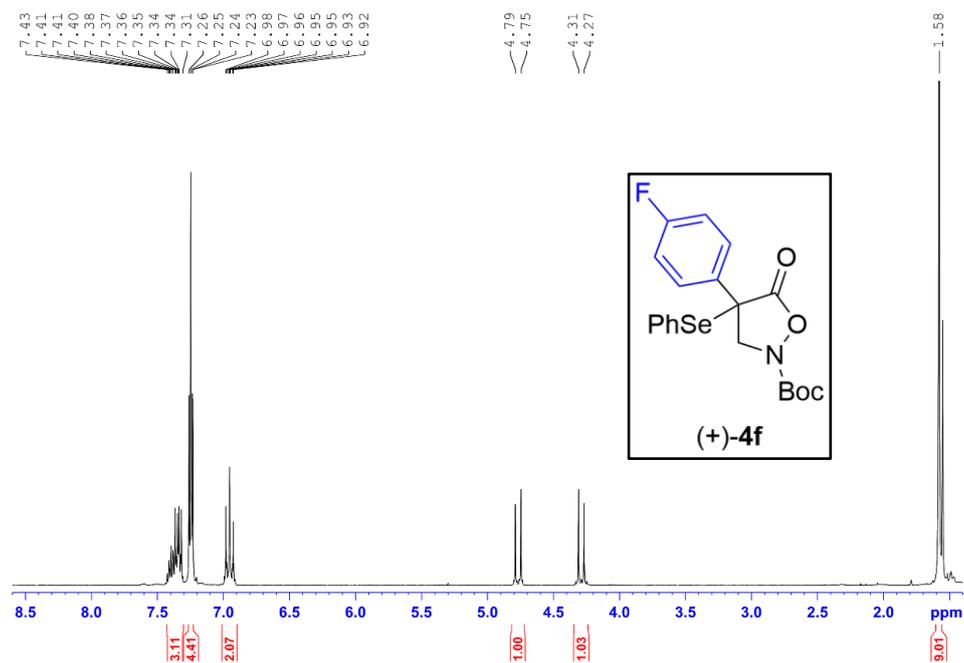
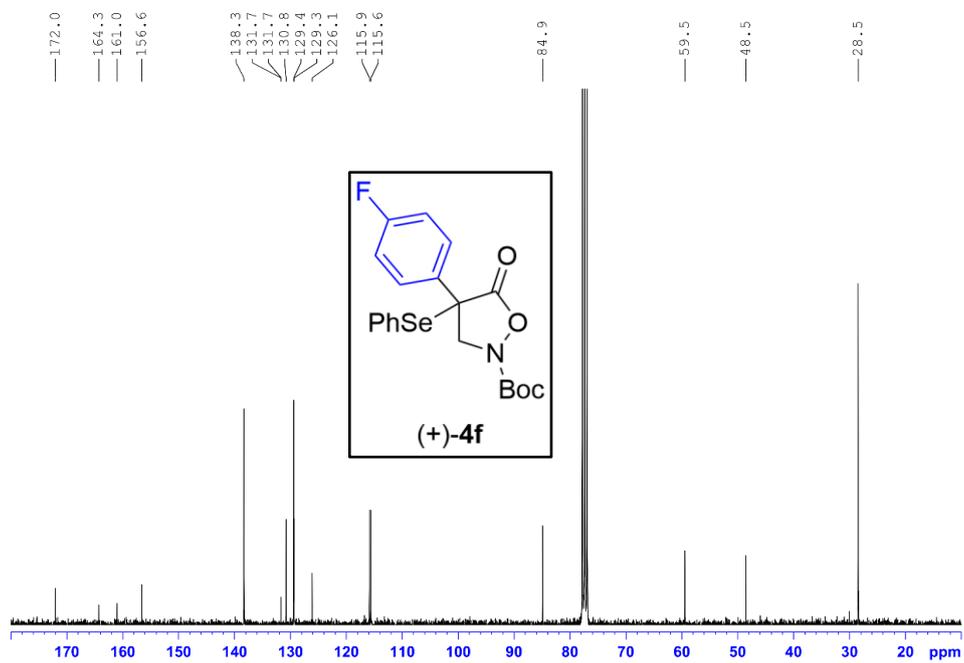
NMR spectra of compound 4a **$^1\text{H-NMR}$ (300 MHz, CDCl_3 , 298 K)** **$^{13}\text{C-NMR}$ (75 MHz, CDCl_3 , 298 K)**

NMR spectra of compound 4b **$^1\text{H-NMR}$ (300 MHz, CDCl_3 , 298 K)** **$^{13}\text{C-NMR}$ (75 MHz, CDCl_3 , 298 K)**

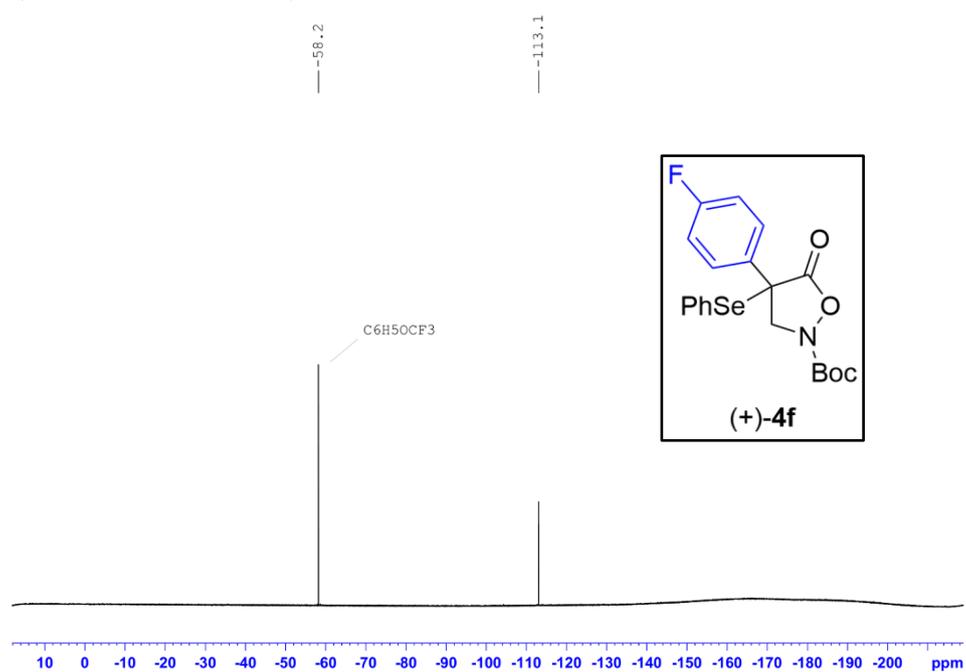
NMR spectra of compound 4c **$^1\text{H-NMR}$ (300 MHz, CDCl_3 , 298 K)** **$^{13}\text{C-NMR}$ (75 MHz, CDCl_3 , 298 K)**

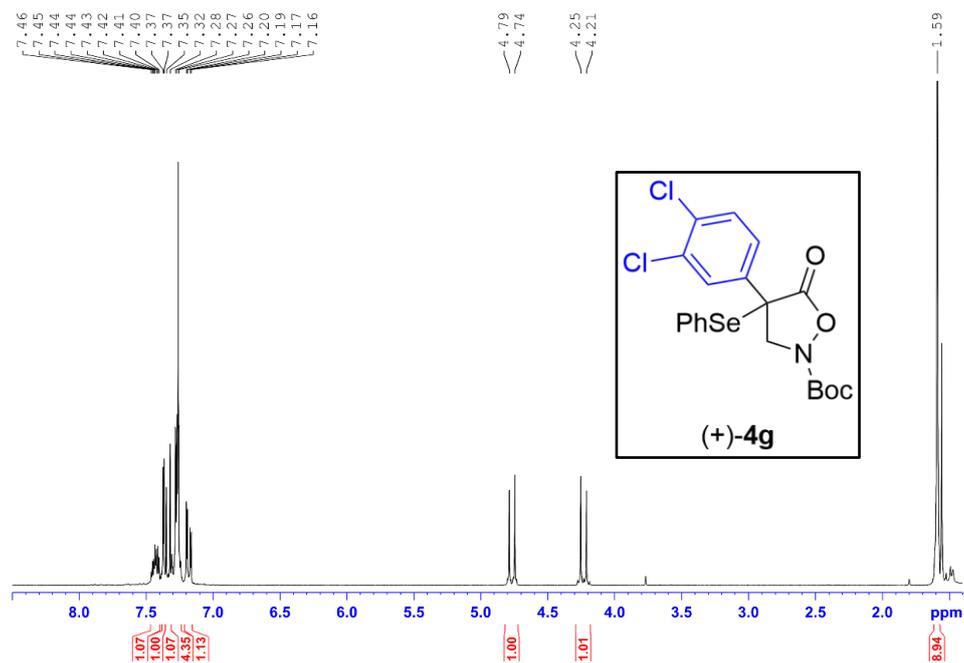
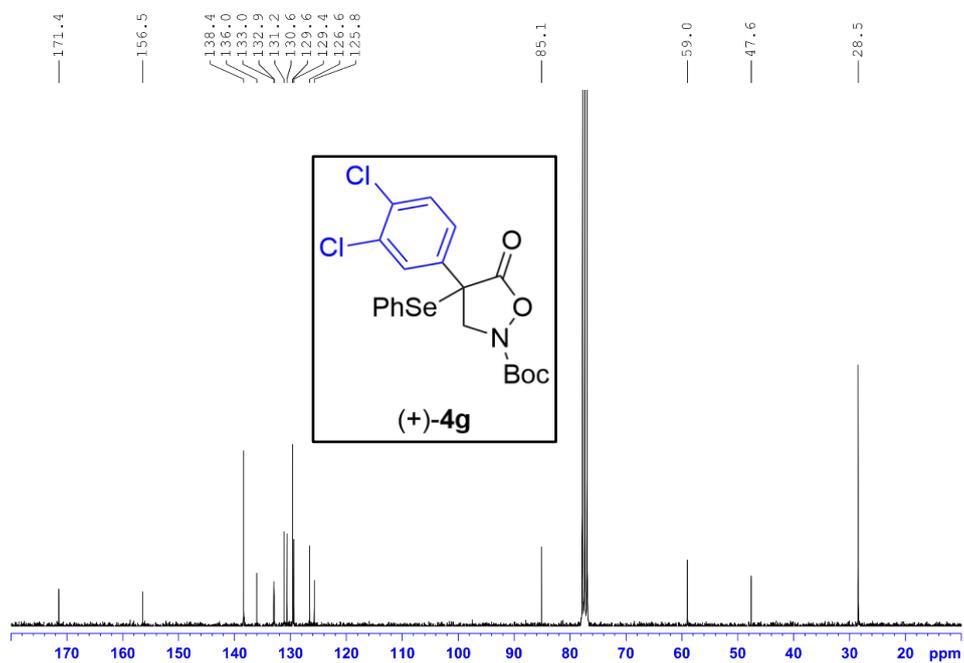
NMR spectra of compound 4d **$^1\text{H-NMR}$ (300 MHz, CDCl_3 , 298 K)** **$^{13}\text{C-NMR}$ (75 MHz, CDCl_3 , 298 K)**

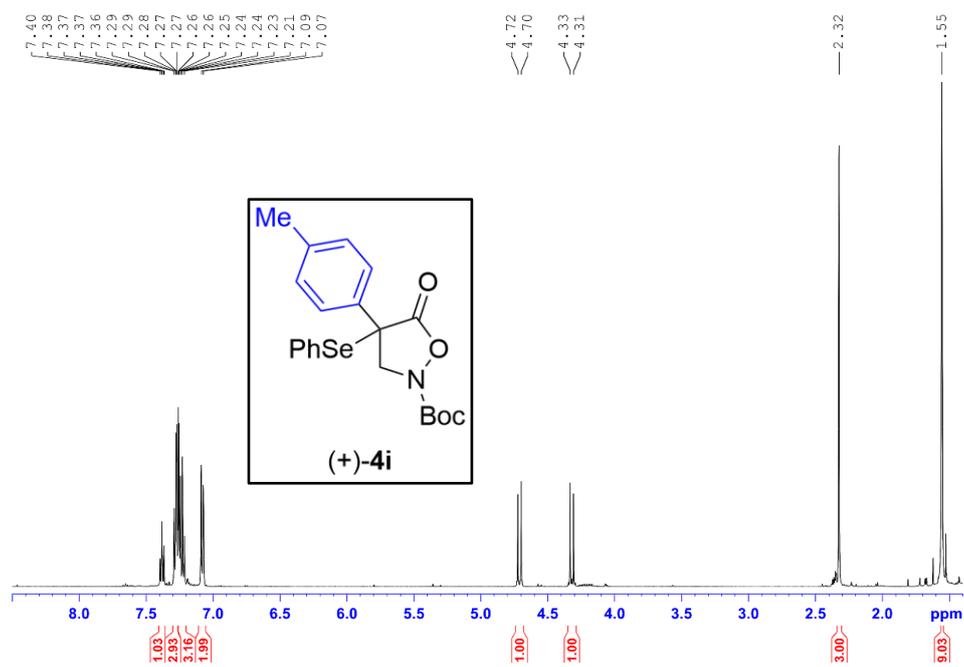
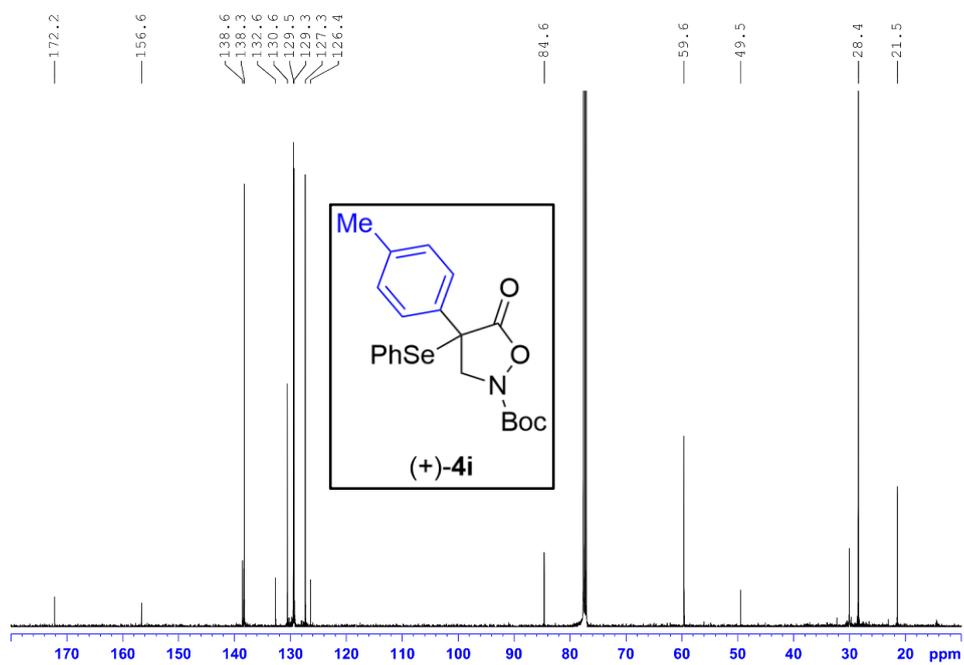
NMR spectra of compound 4e **$^1\text{H-NMR}$ (300 MHz, CDCl_3 , 298 K)** **$^{13}\text{C-NMR}$ (75 MHz, CDCl_3 , 298 K)**

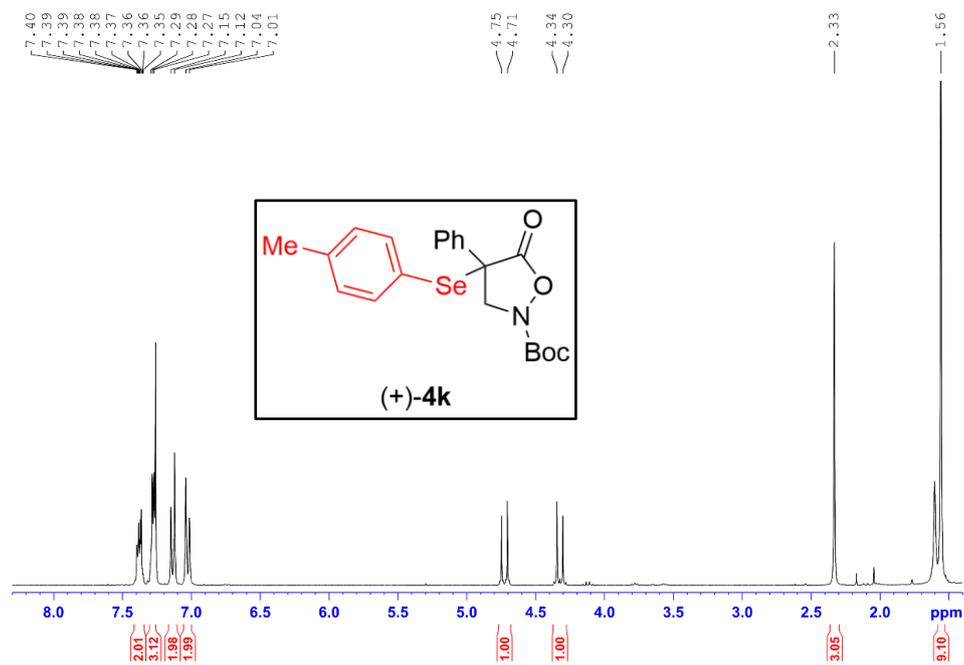
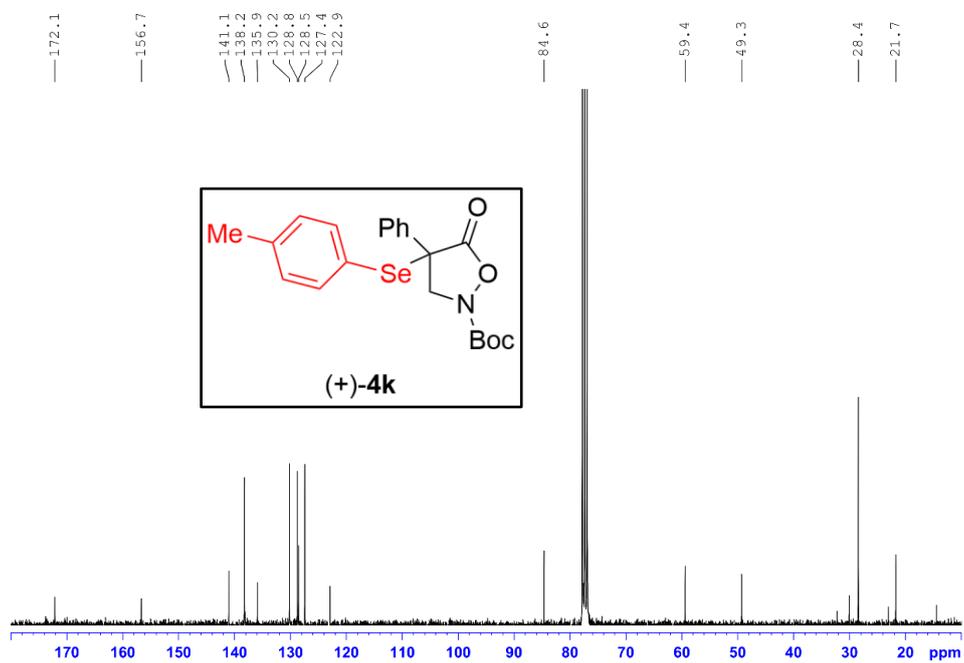
NMR spectra of compound 4f **$^1\text{H-NMR}$ (300 MHz, CDCl_3 , 298 K)** **$^{13}\text{C-NMR}$ (75 MHz, CDCl_3 , 298 K)**

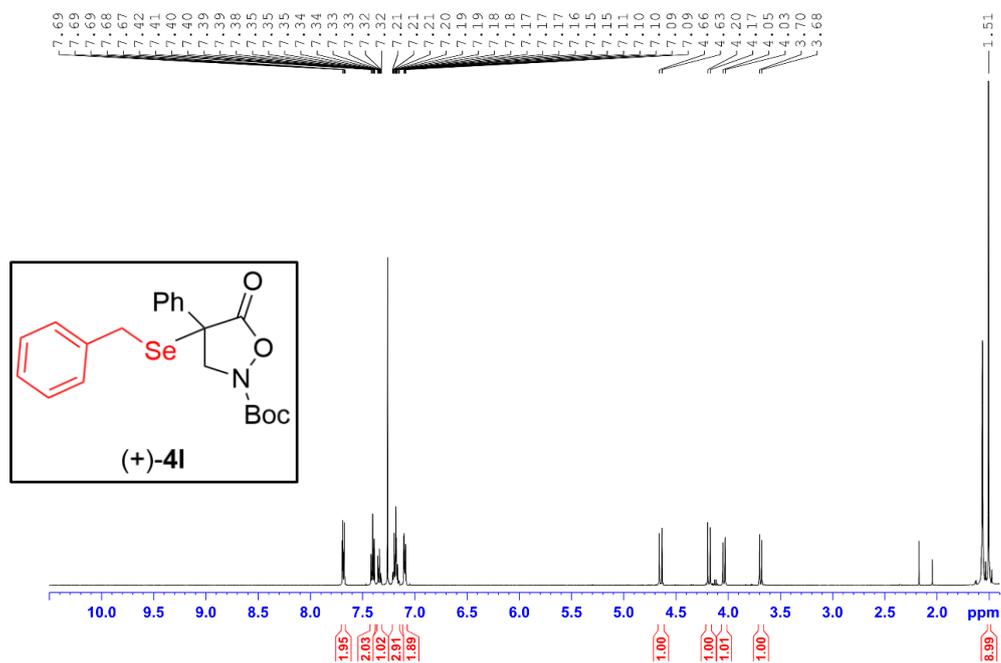
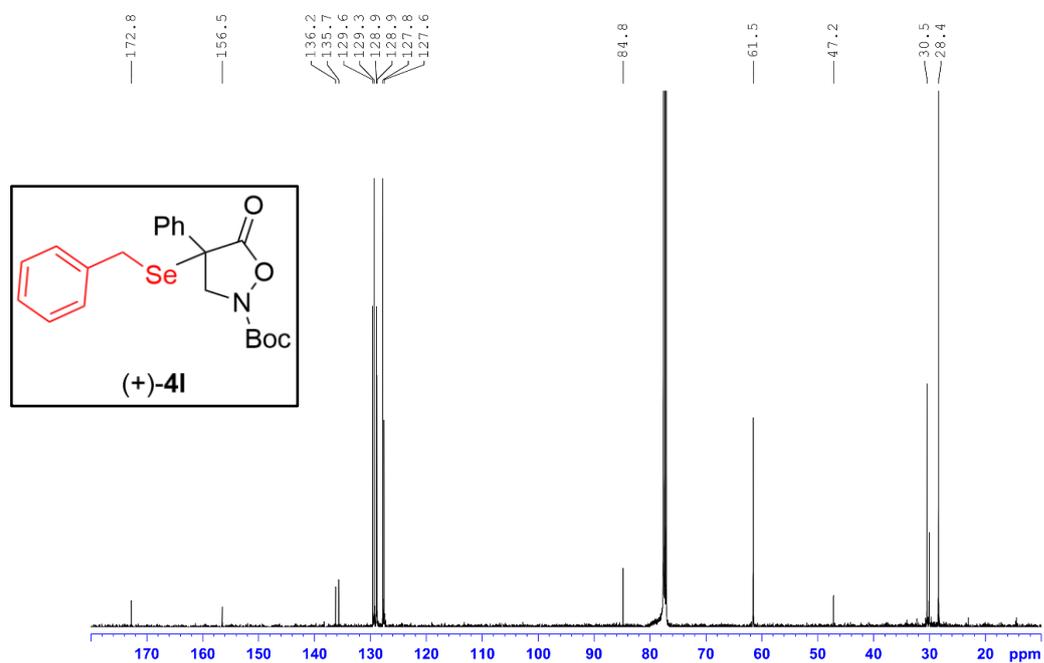
^{19}F -NMR (282 MHz, CDCl_3 , 298 K)

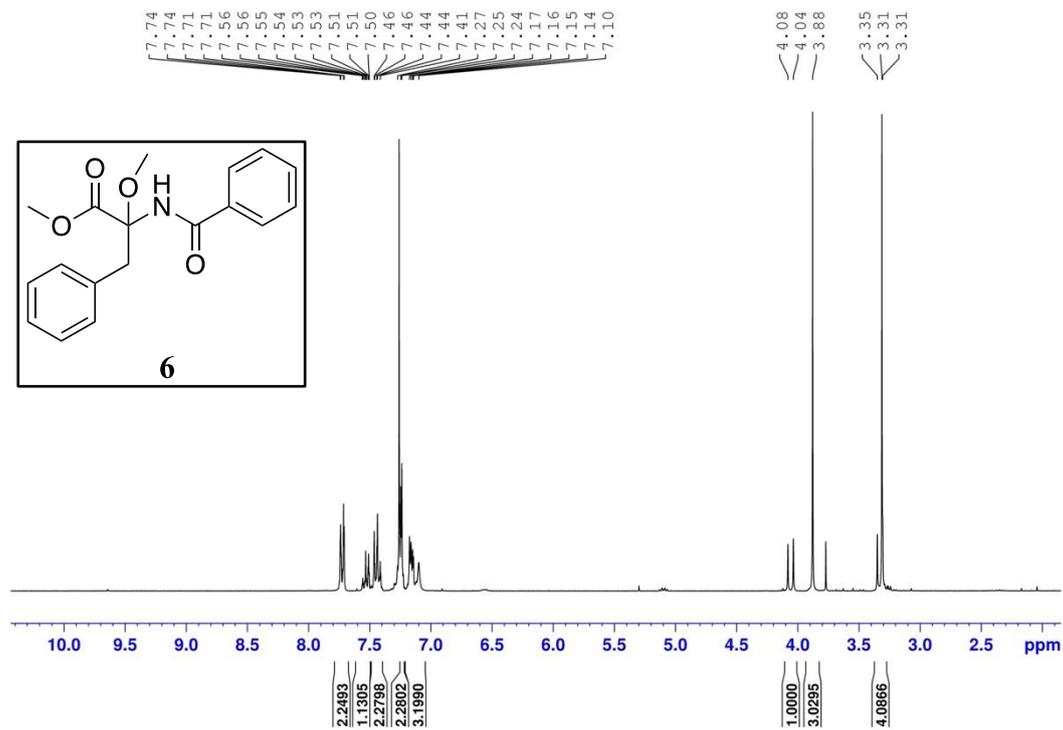
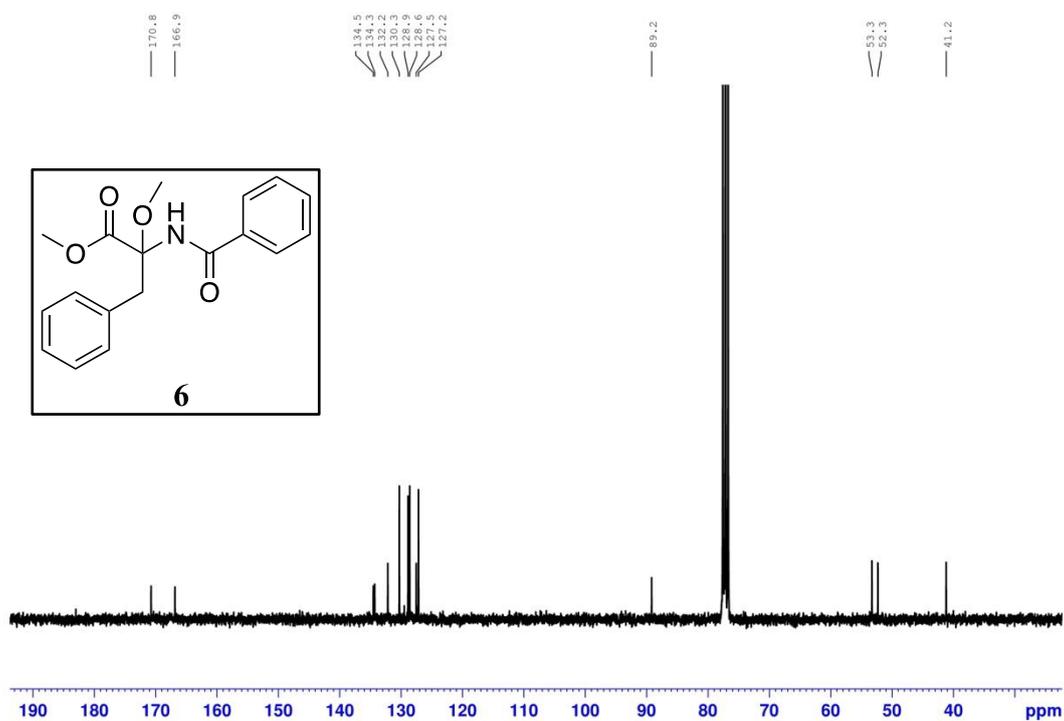


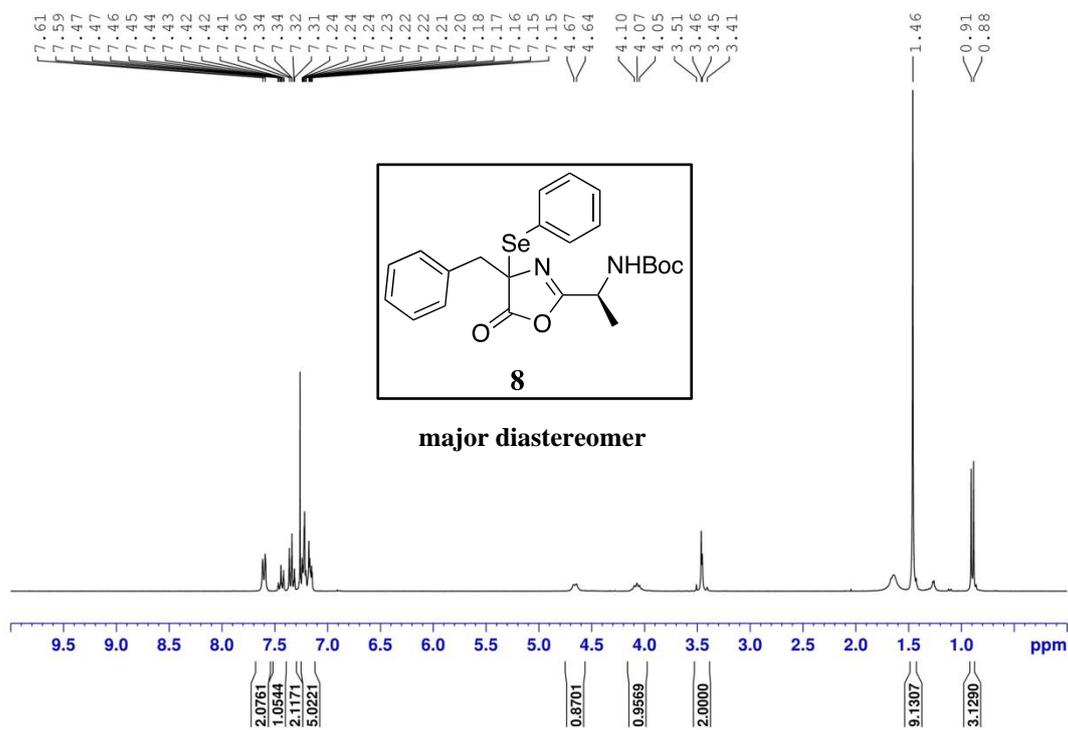
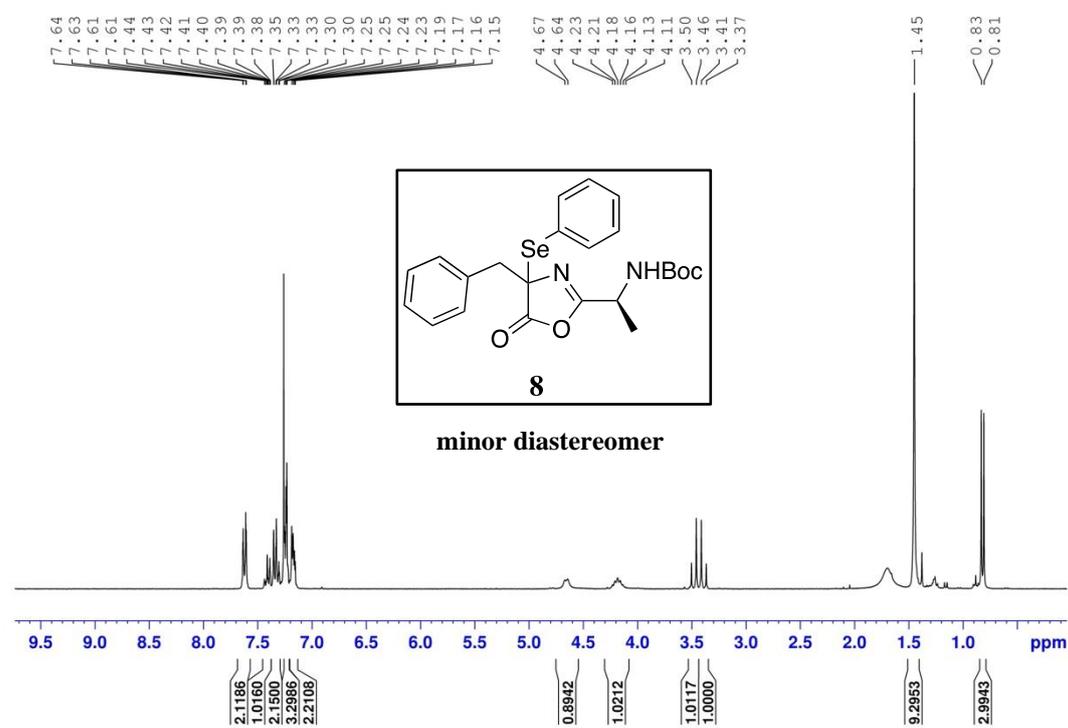
NMR spectra of compound **4g** **$^1\text{H-NMR}$ (300 MHz, CDCl_3 , 298 K)** **$^{13}\text{C-NMR}$ (75 MHz, CDCl_3 , 298 K)**

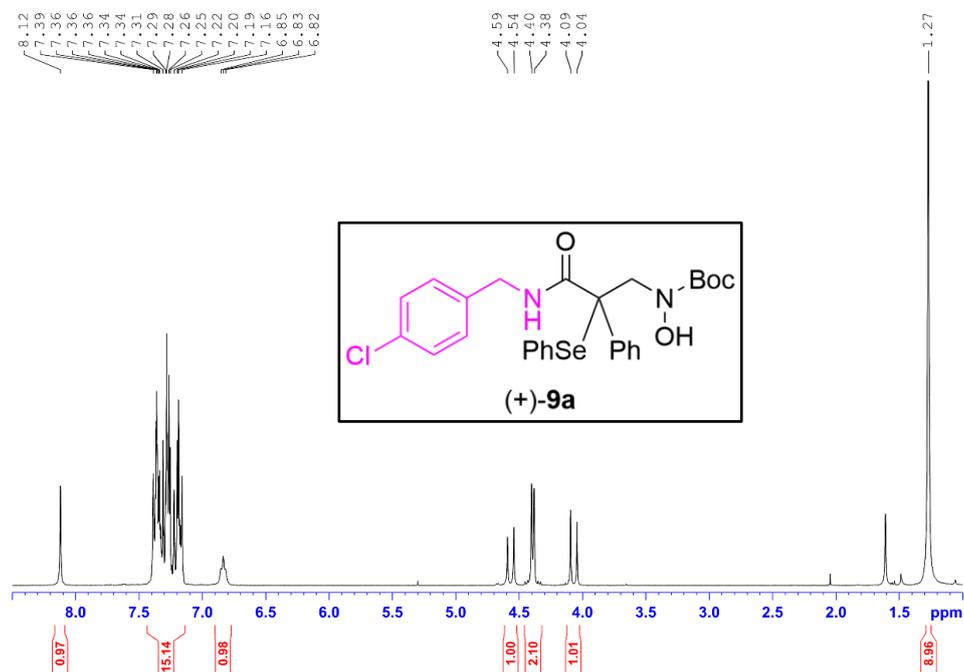
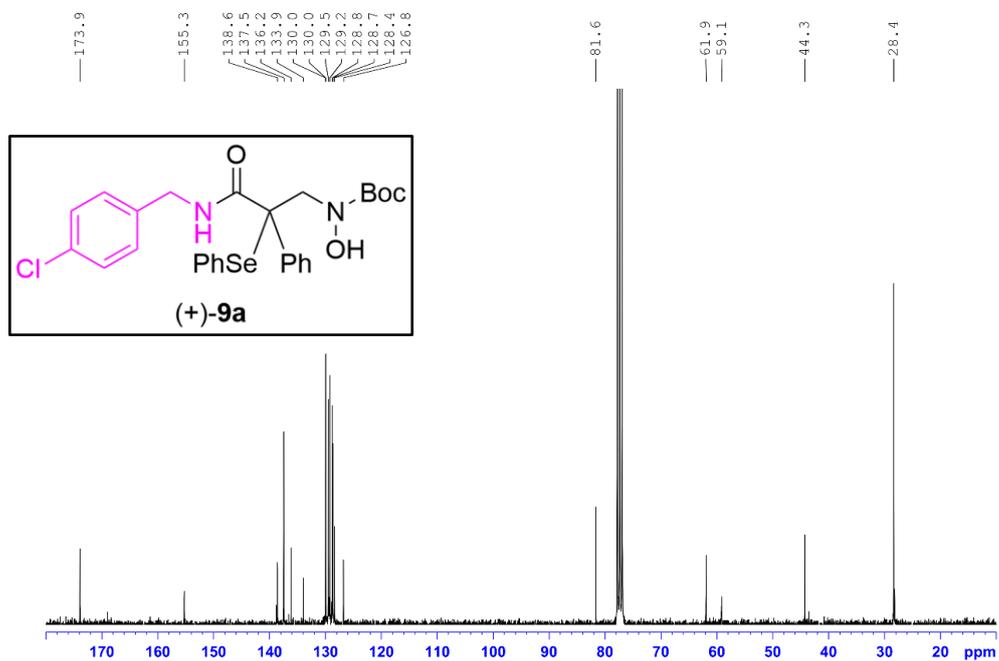
NMR spectra of compound **4i** **$^1\text{H-NMR}$ (500 MHz, CDCl_3 , 298 K)** **$^{13}\text{C-NMR}$ (126 MHz, CDCl_3 , 298 K)**

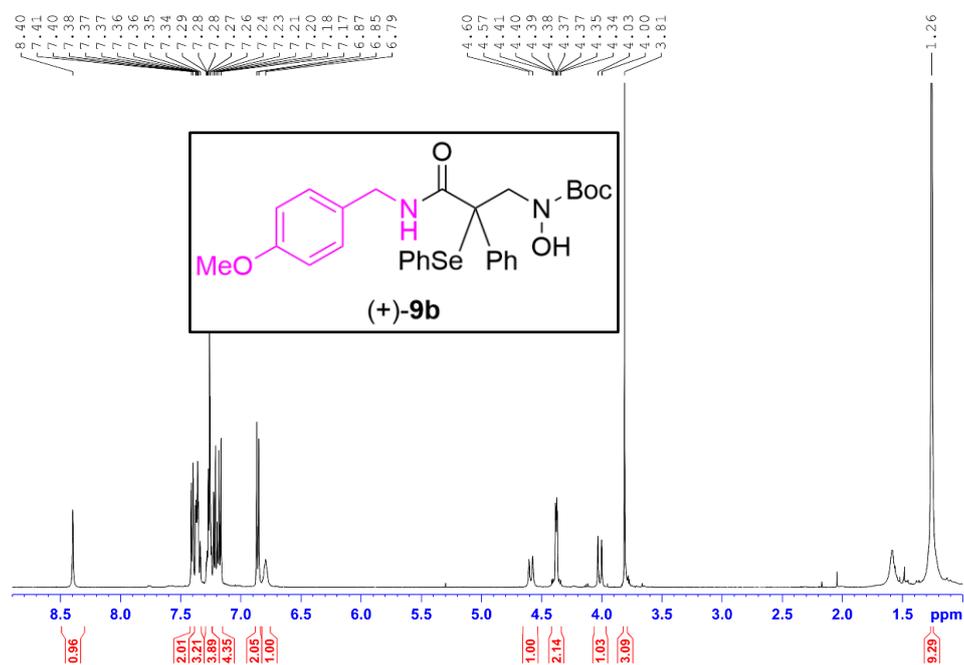
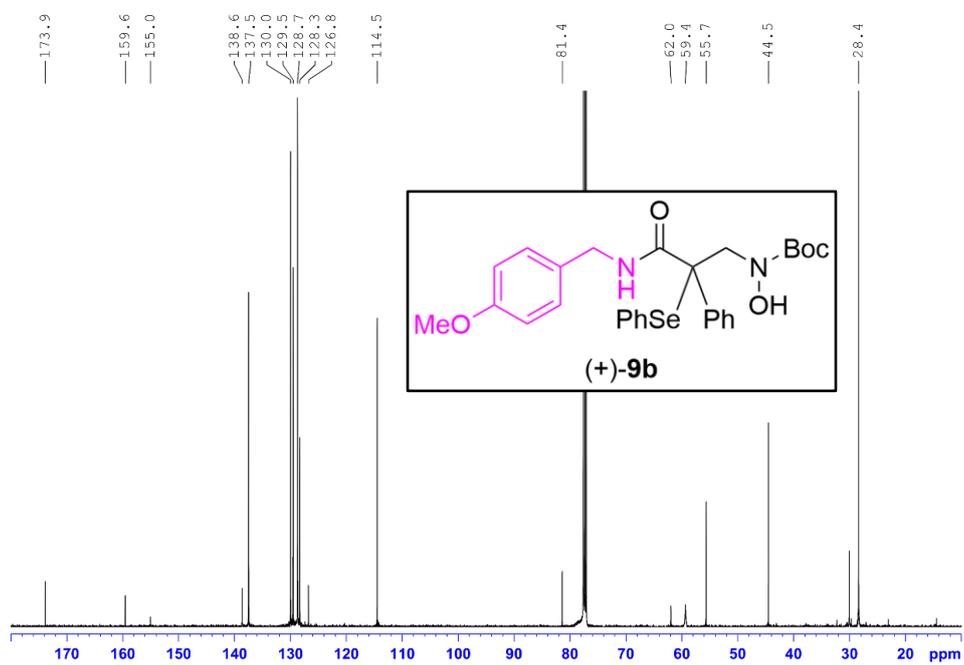
NMR spectra of compound 4k **$^1\text{H-NMR}$ (300 MHz, CDCl_3 , 298 K)** **$^{13}\text{C-NMR}$ (75 MHz, CDCl_3 , 298 K)**

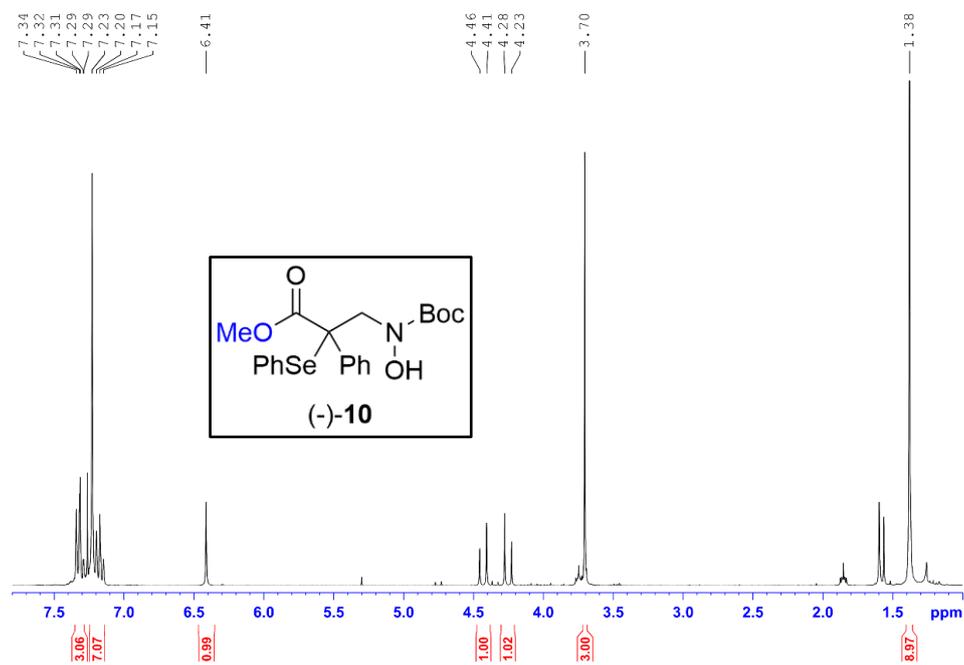
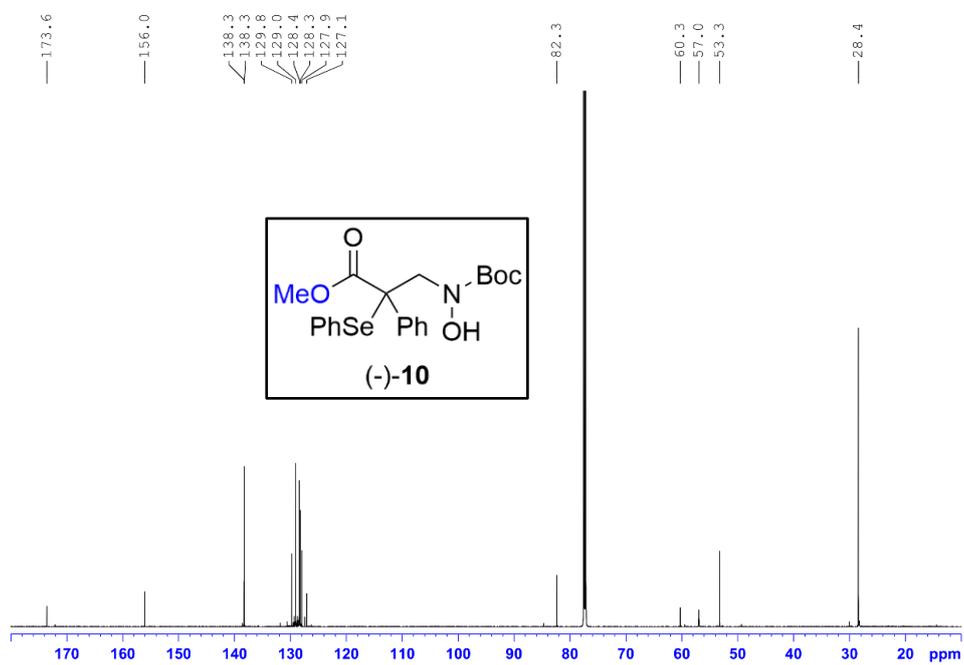
NMR spectra of compound 4I **$^1\text{H-NMR}$ (500 MHz, CDCl_3 , 298 K)** **$^{13}\text{C-NMR}$ (126 MHz, CDCl_3 , 298 K)**

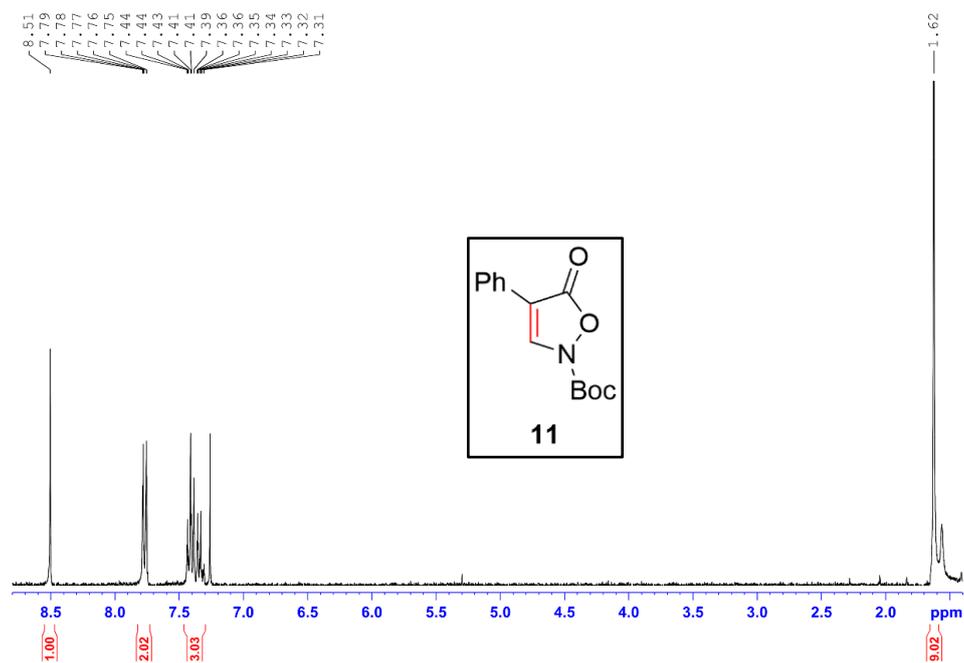
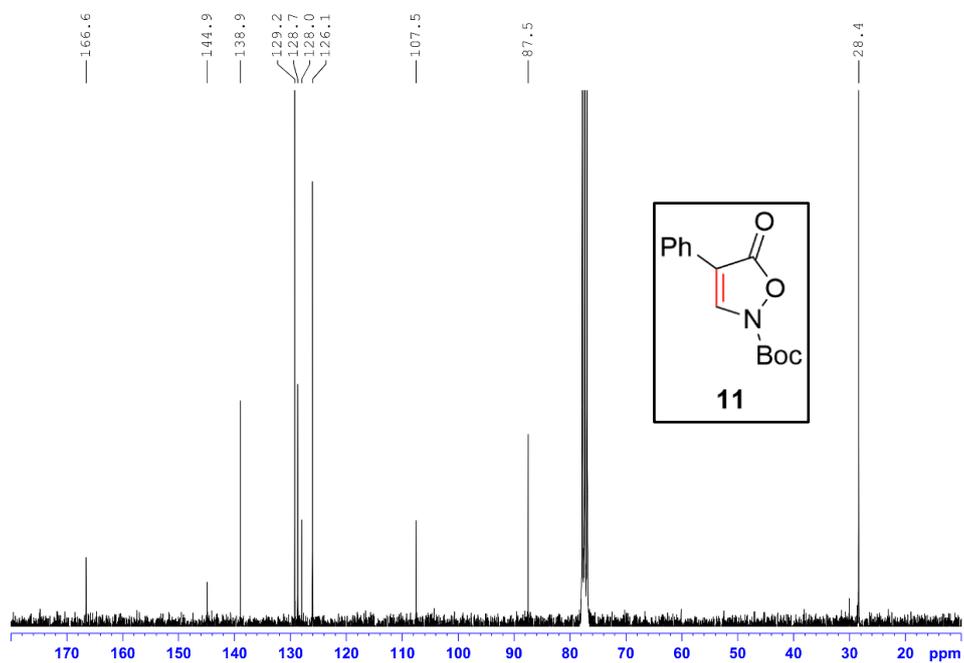
NMR spectra of compound 6 **$^1\text{H-NMR}$ (300 MHz, CDCl_3 , 298 K)** **$^{13}\text{C-NMR}$ (75 MHz, CDCl_3 , 298 K)**

NMR spectra of compound 8 **$^1\text{H-NMR}$ (300 MHz, CDCl_3 , 298 K)** **$^1\text{H-NMR}$ (300 MHz, CDCl_3 , 298 K)**

NMR spectra of compound 9a **$^1\text{H-NMR}$ (300 MHz, CDCl_3 , 298 K)** **$^{13}\text{C-NMR}$ (75 MHz, CDCl_3 , 298 K)**

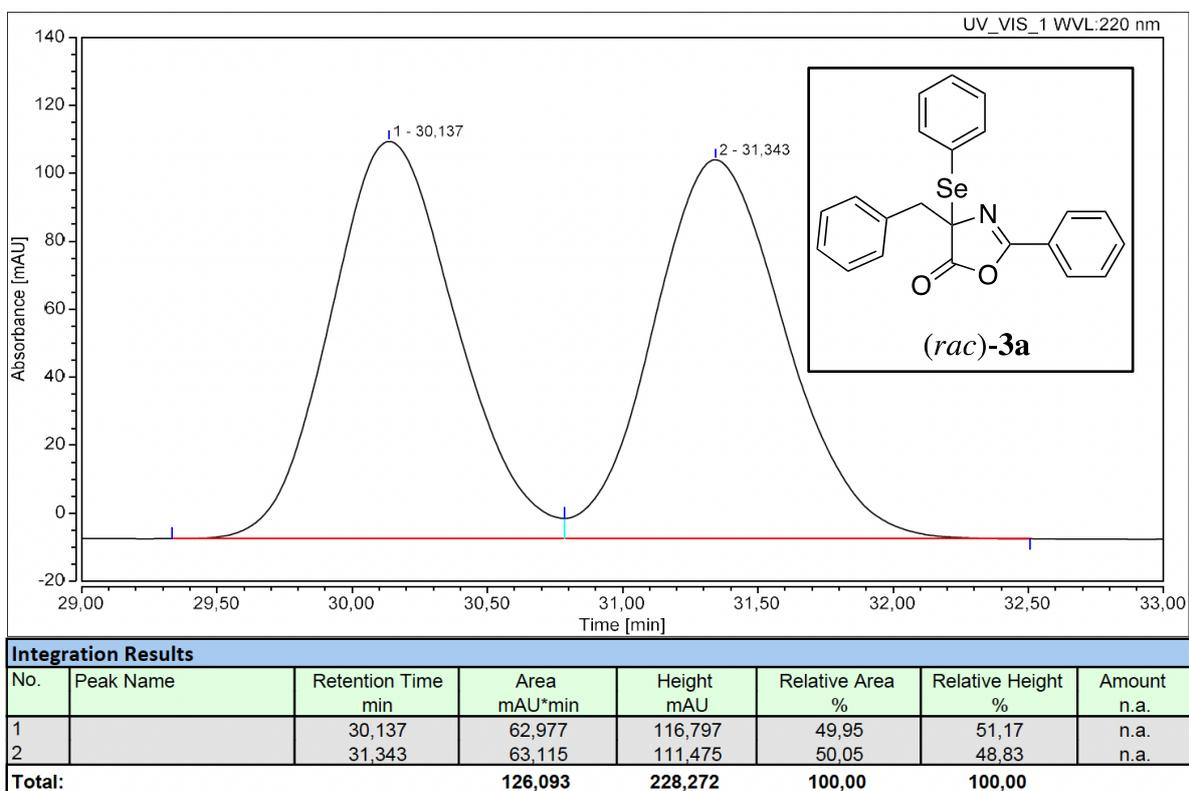
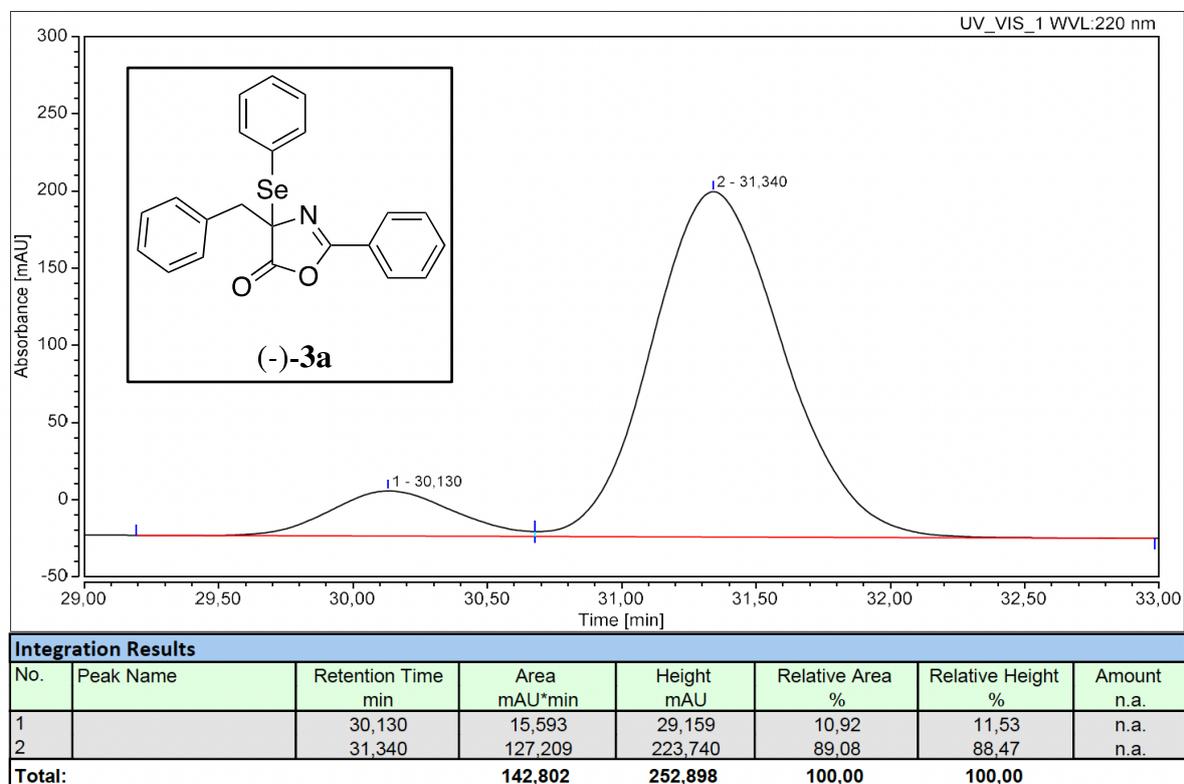
NMR spectra of compound 9b **$^1\text{H-NMR}$ (500 MHz, CDCl_3 , 298 K)** **$^{13}\text{C-NMR}$ (126 MHz, CDCl_3 , 298 K)**

NMR spectra of compound 10 **$^1\text{H-NMR}$ (300 MHz, CDCl_3 , 298 K)** **$^{13}\text{C-NMR}$ (176 MHz, CDCl_3 , 298 K)**

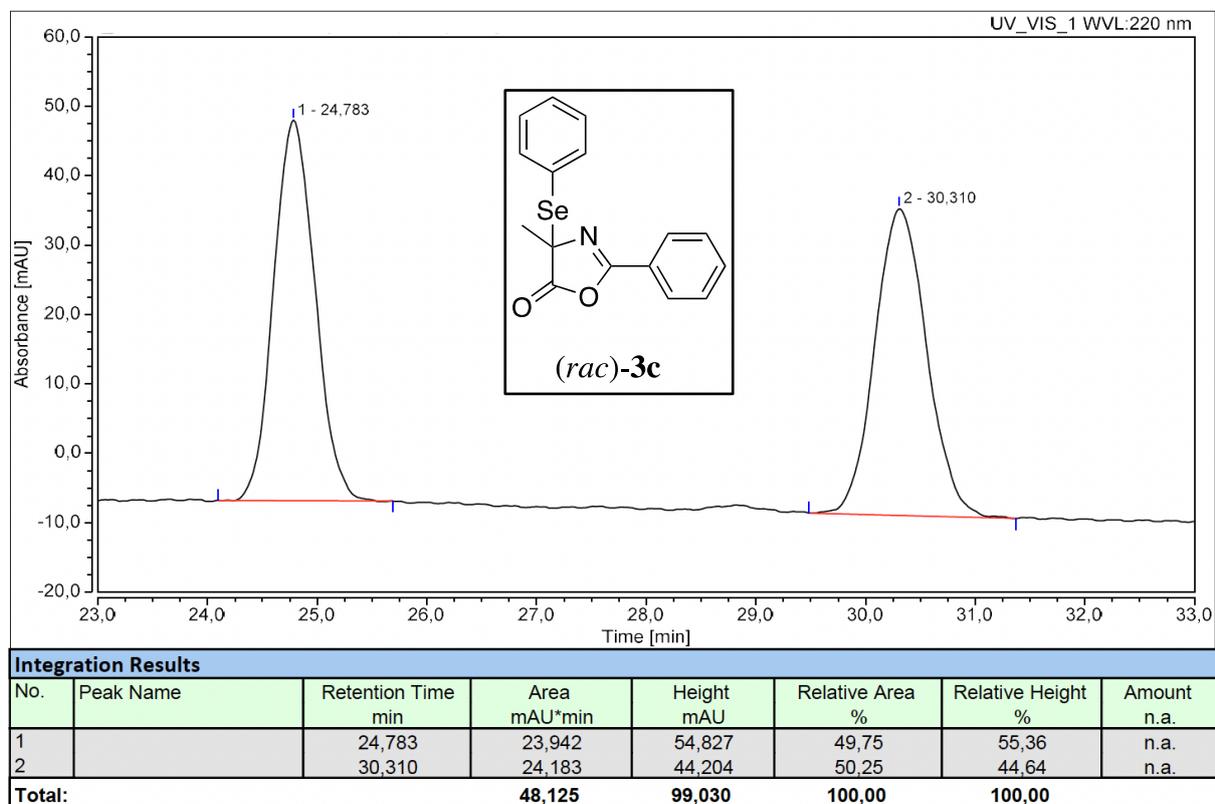
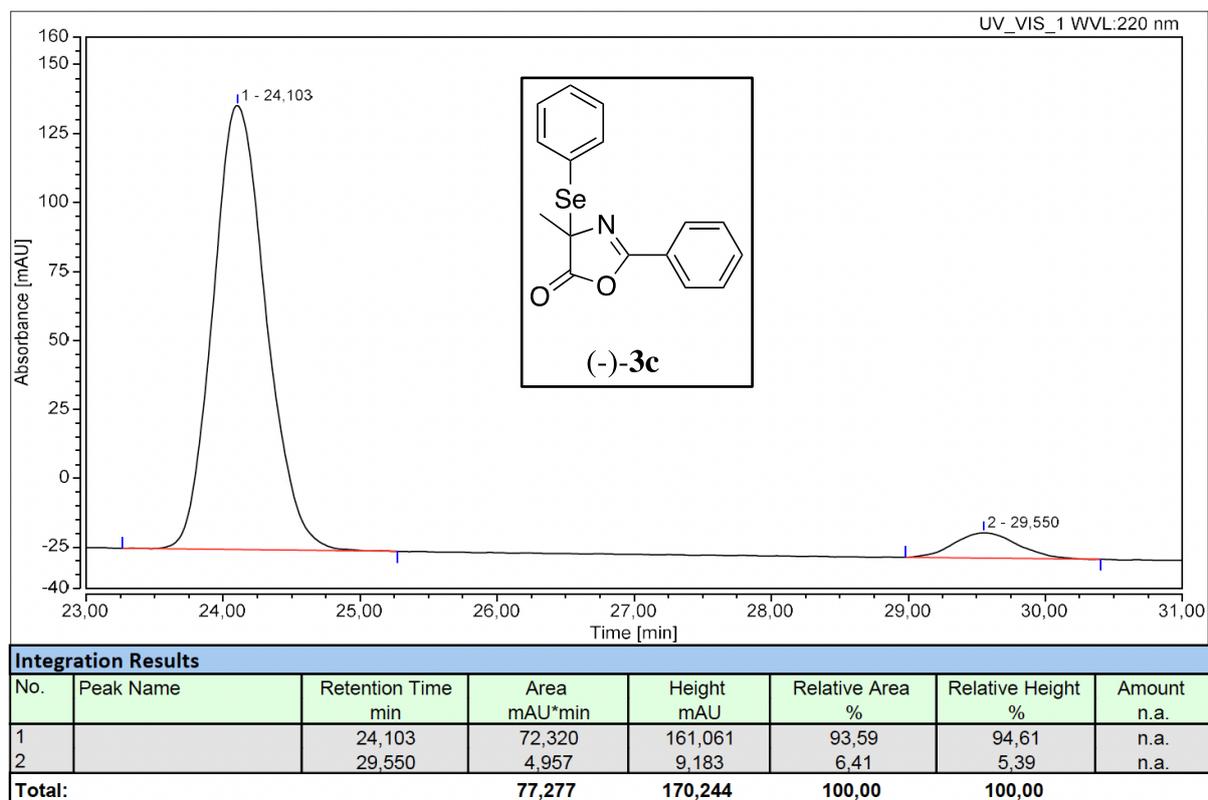
NMR spectra of compound 11 **$^1\text{H-NMR}$ (300 MHz, CDCl_3 , 298 K)** **$^{13}\text{C-NMR}$ (75 MHz, CDCl_3 , 298 K)**

10. HPLC Traces of Racemic and Enantioenriched Selenation Products

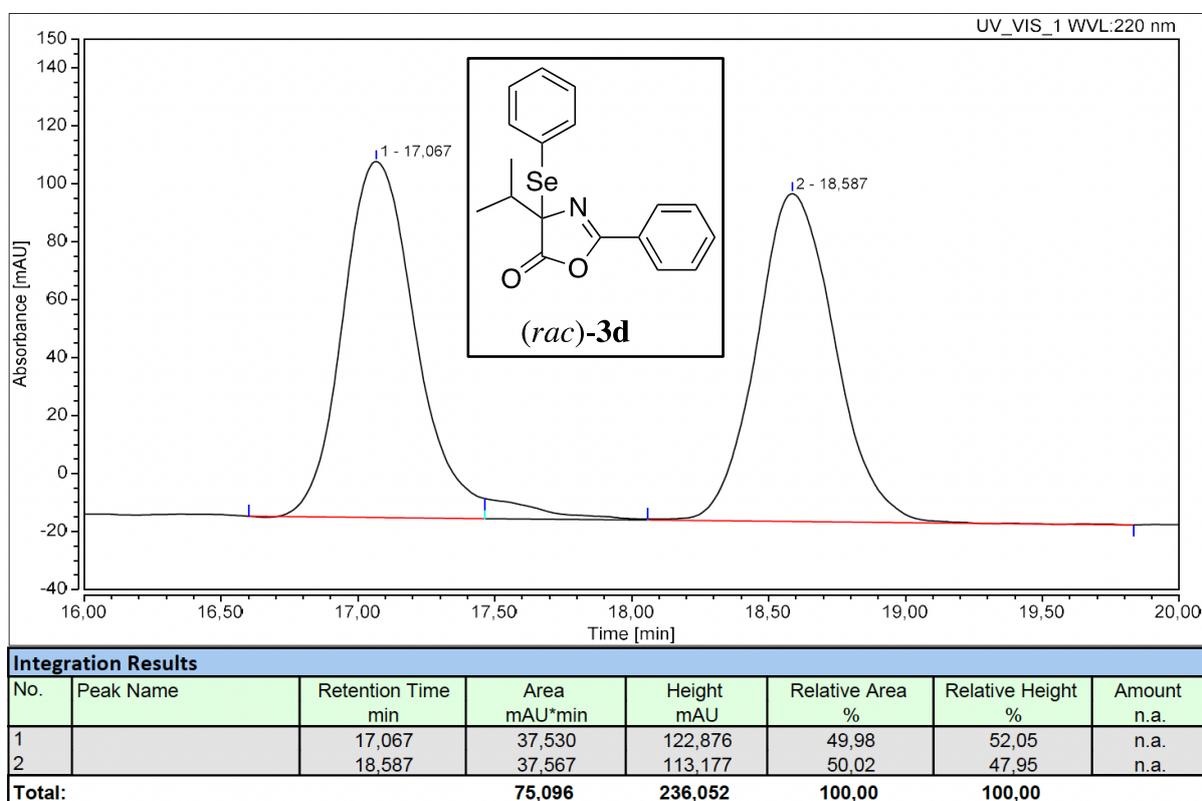
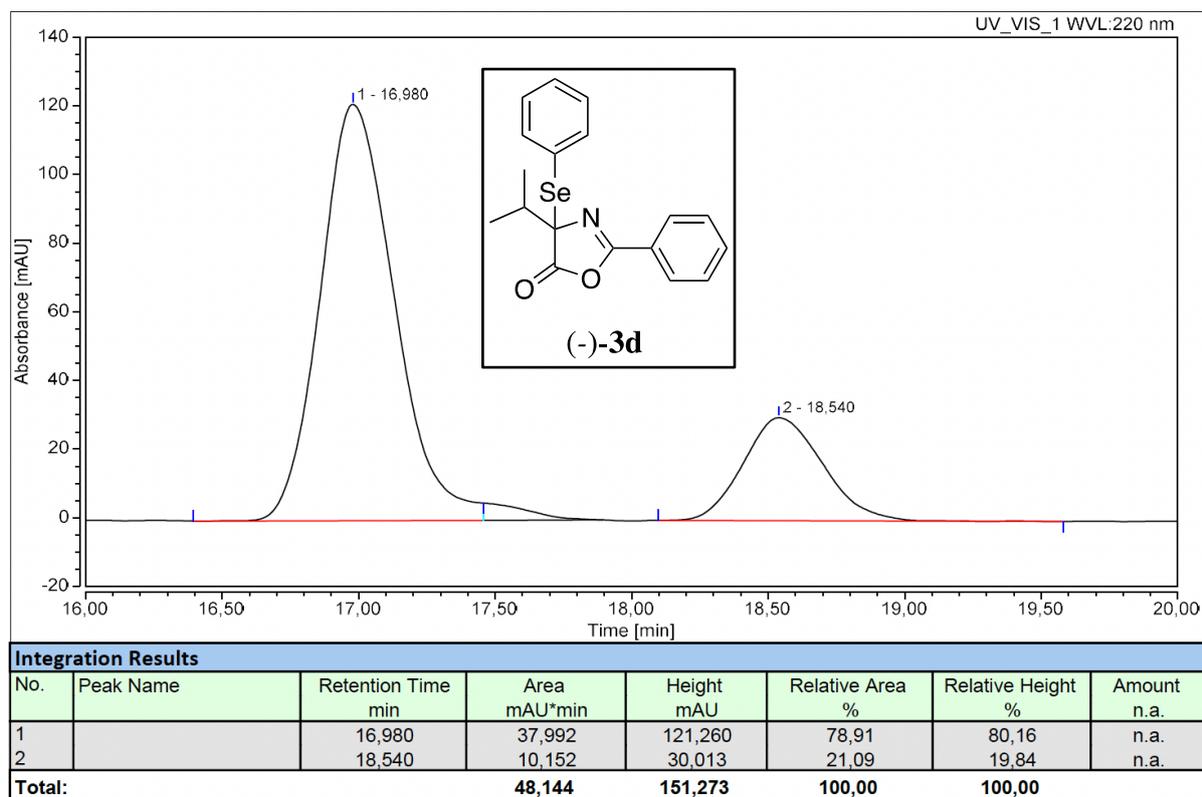
HPLC traces of **3a** (Chiralpak AD-H, *n*-hexane:*i*-PrOH = 20:1, flow rate 0.3 mL/min, 10 °C, $\lambda = 220$ nm)



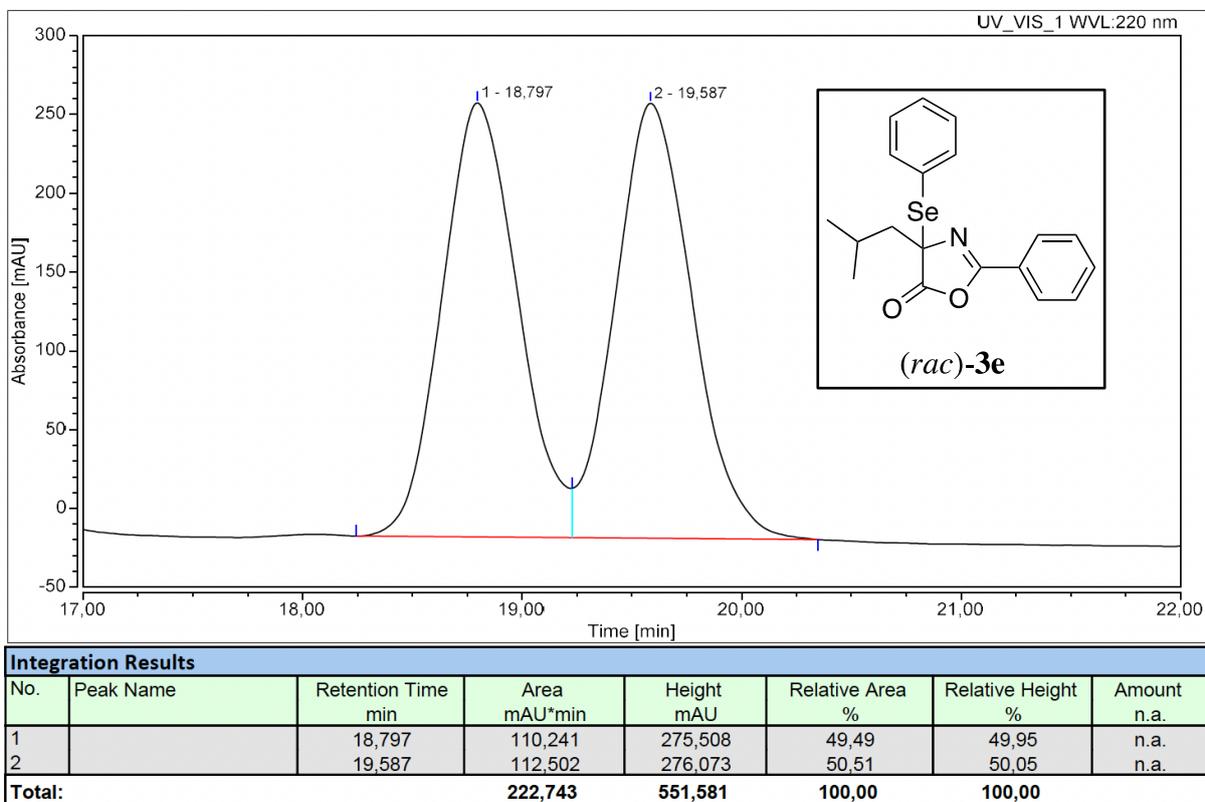
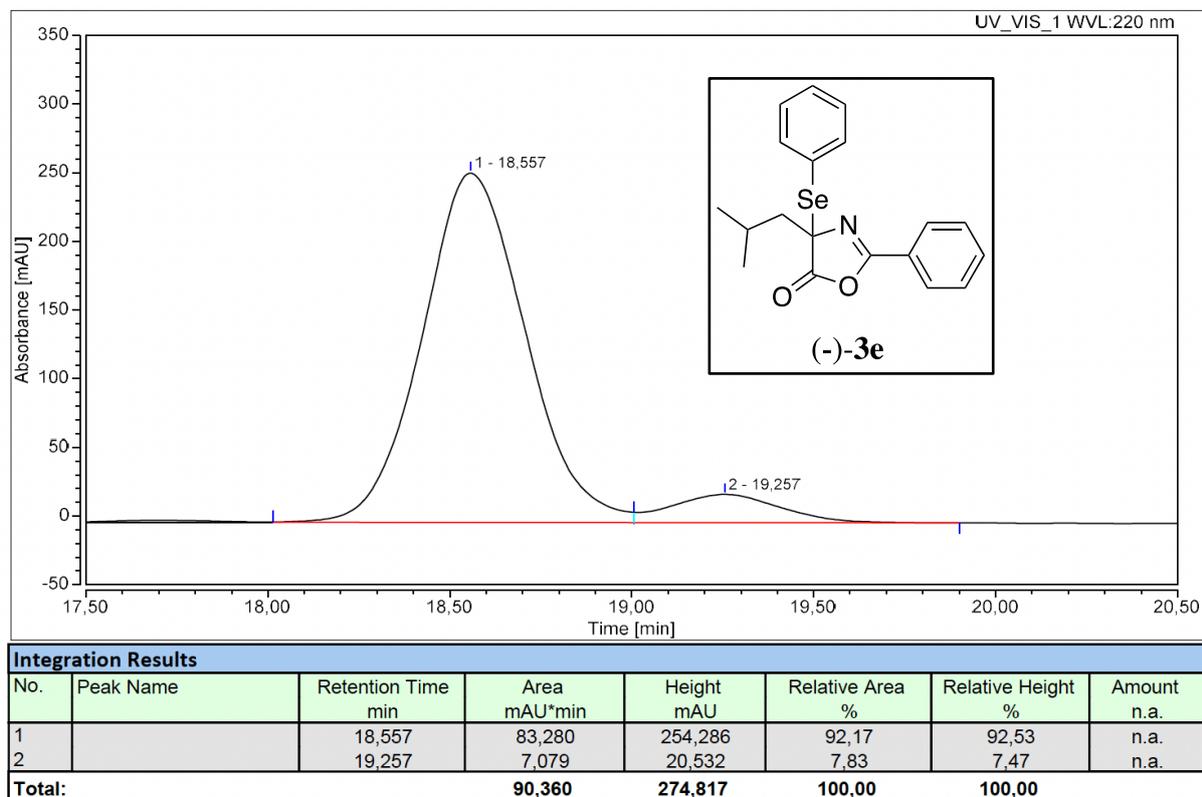
HPLC traces of **3c** (Chiralpak AD-H, *n*-hexane:*i*-PrOH = 20:1, flow rate 0.3 mL/min, 10 °C, λ = 220 nm)



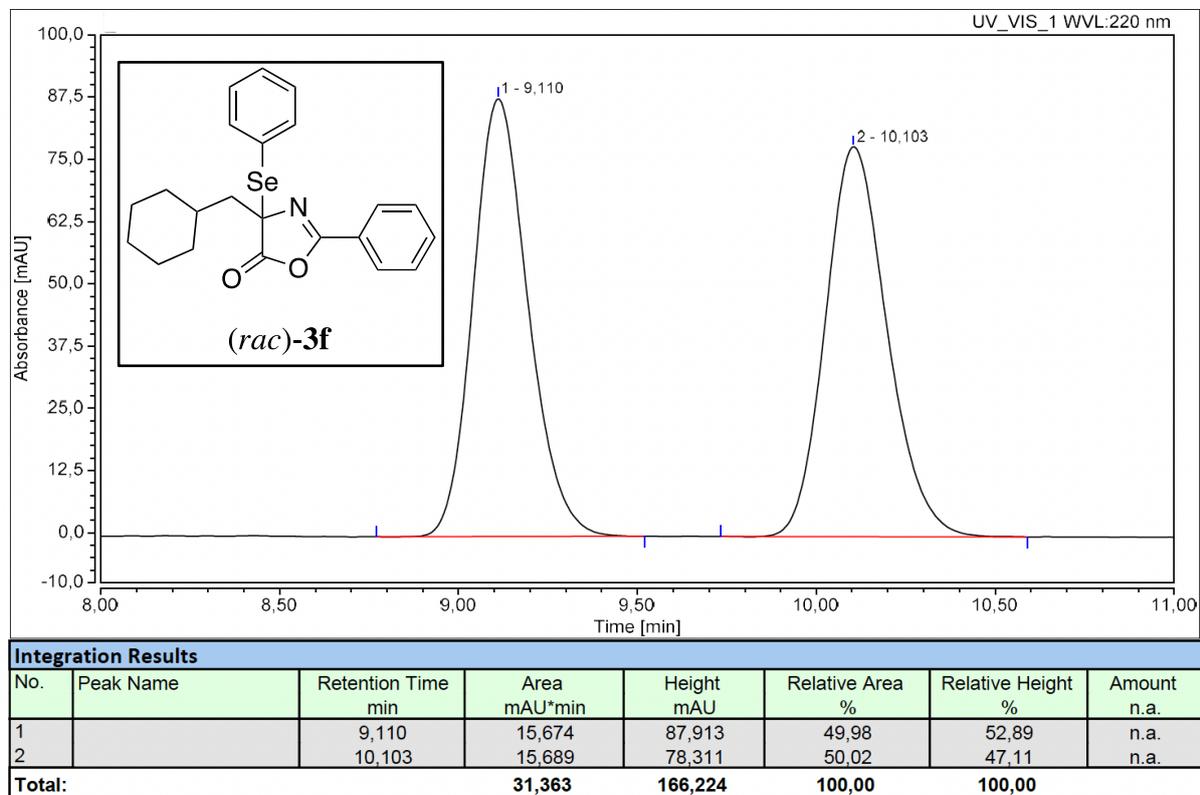
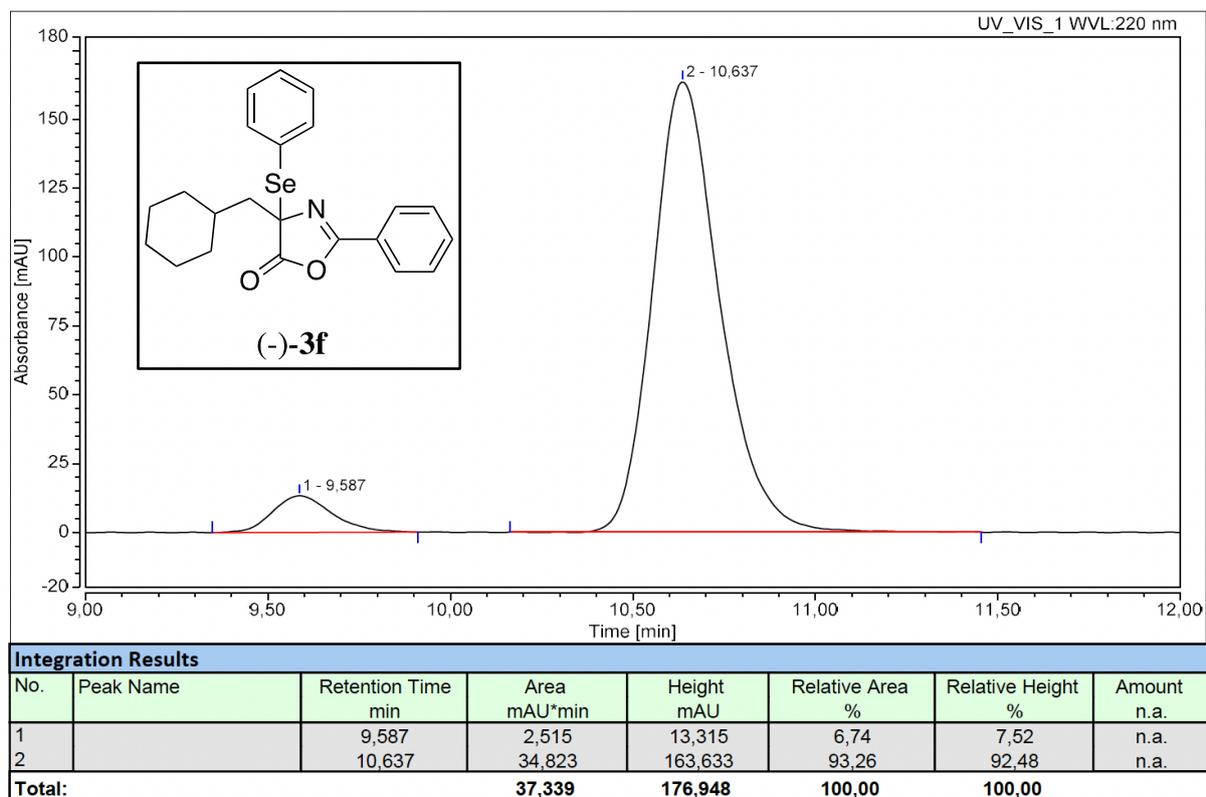
HPLC traces of **3d** (Chiralpak AD-H, *n*-hexane:*i*-PrOH = 20:1, flow rate 0.3 mL/min, 10 °C, $\lambda = 220$ nm)



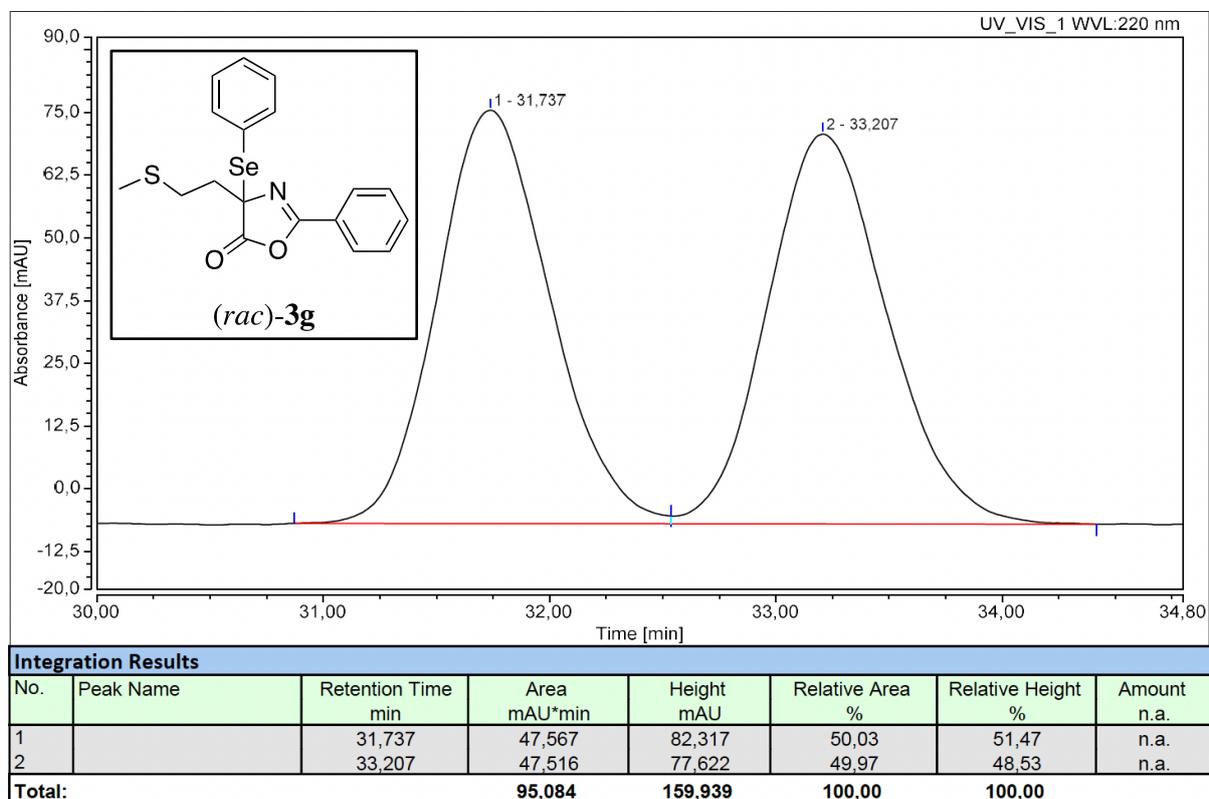
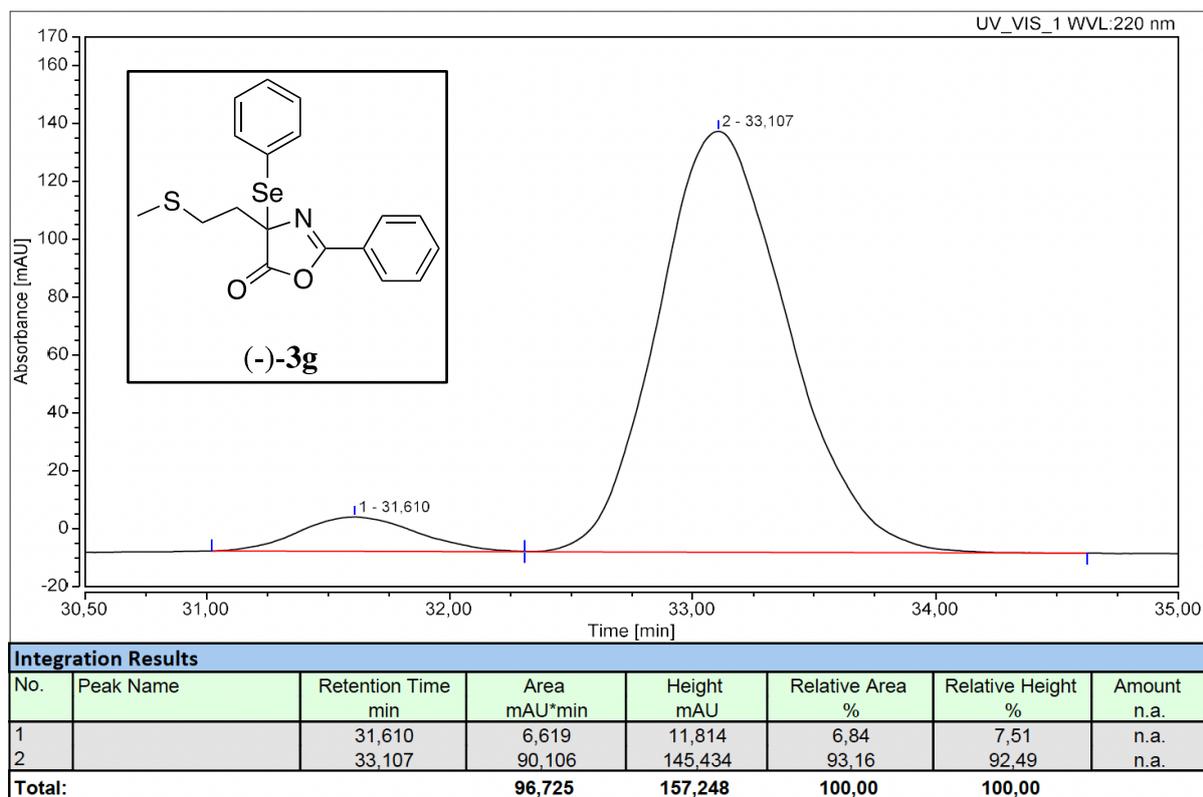
HPLC traces of **3e** (YMC Chiral Art Cellulose SB, *n*-hexane:*i*-PrOH = 80:1, flow rate 0.2 mL/min, 10 °C, λ = 220 nm)



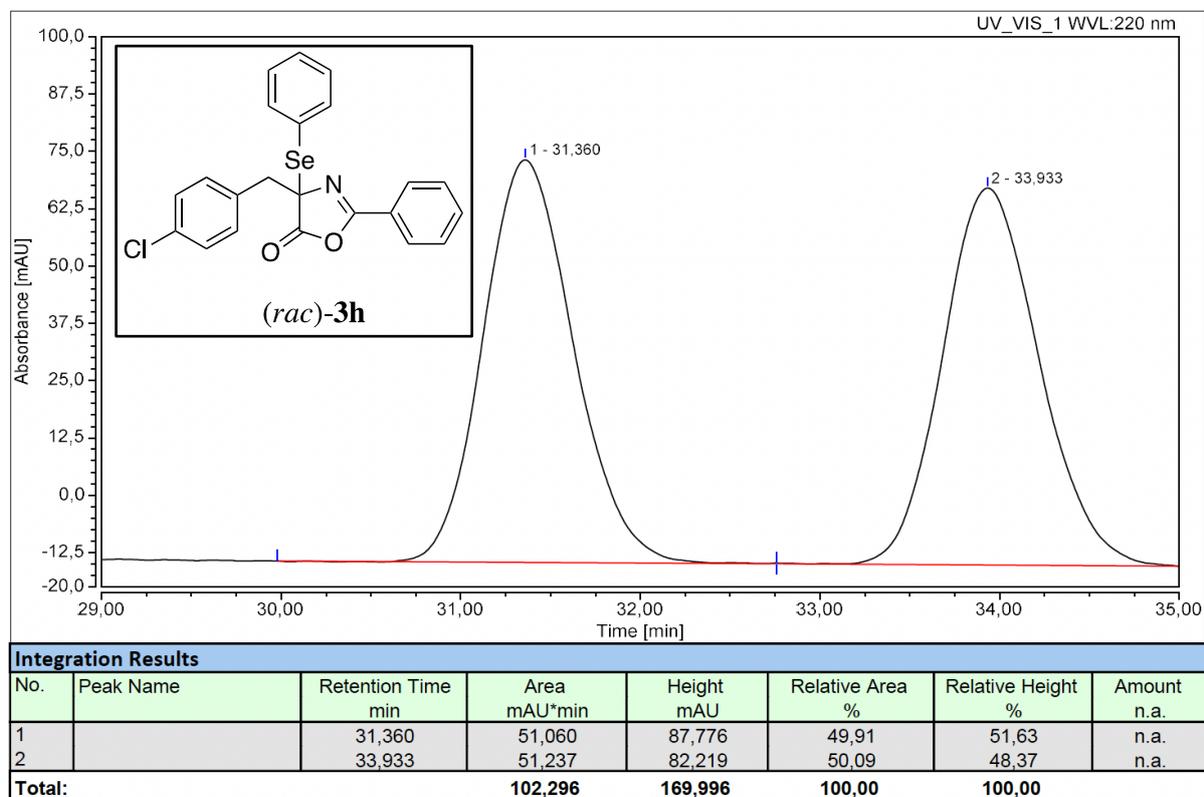
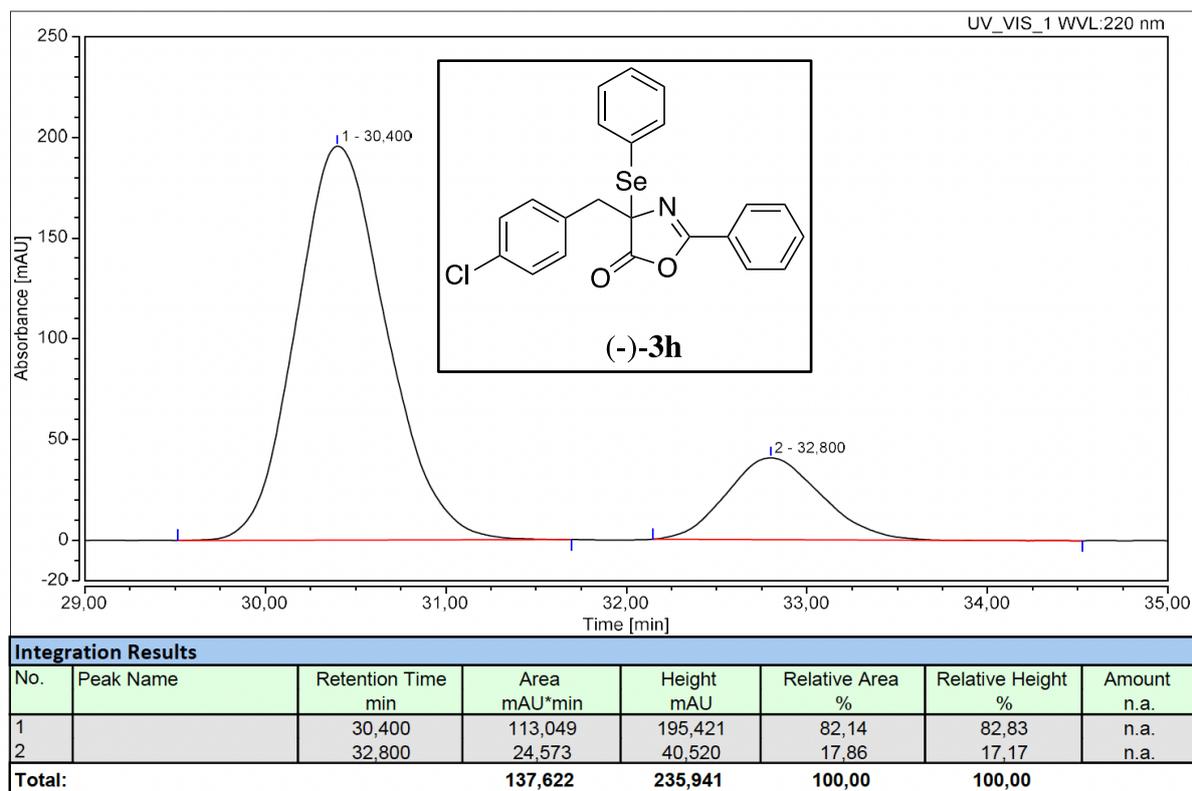
HPLC traces of **3f** (Chiralpak AD-H, *n*-hexane:*i*-PrOH = 20:1, flow rate 0.7 mL/min, 10 °C, $\lambda = 220$ nm)



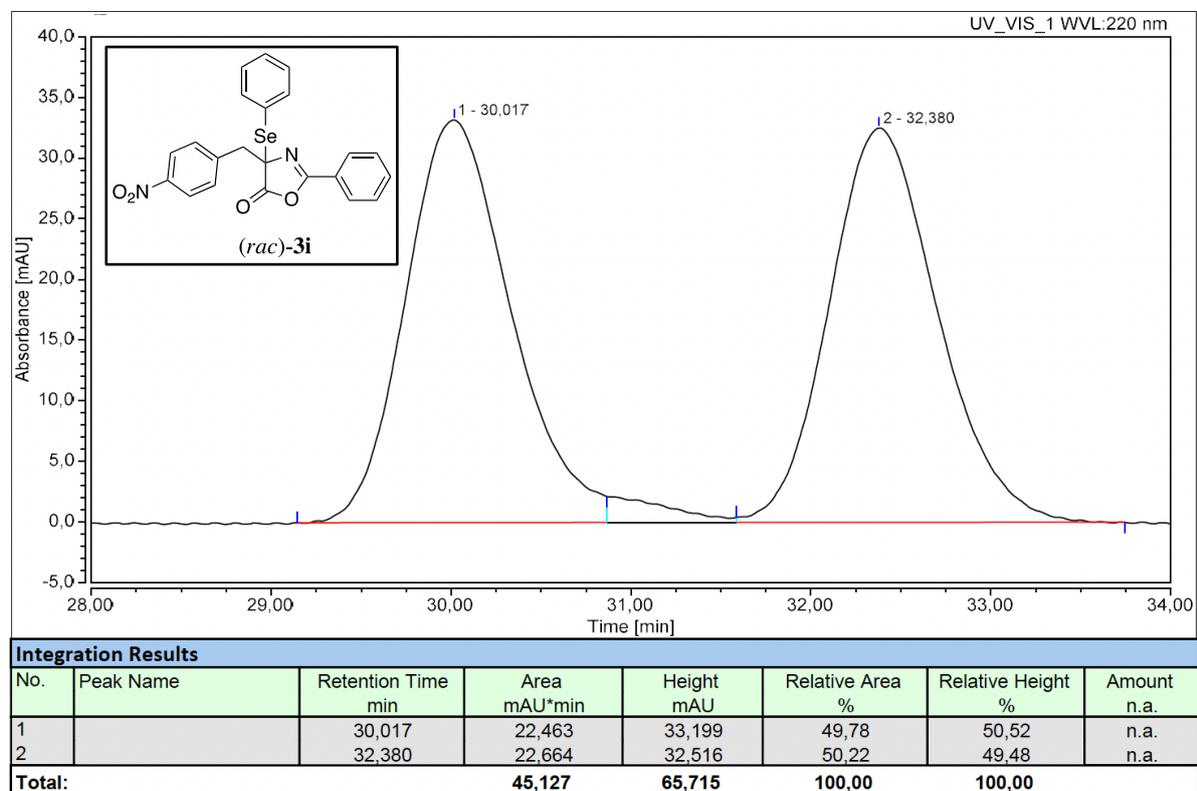
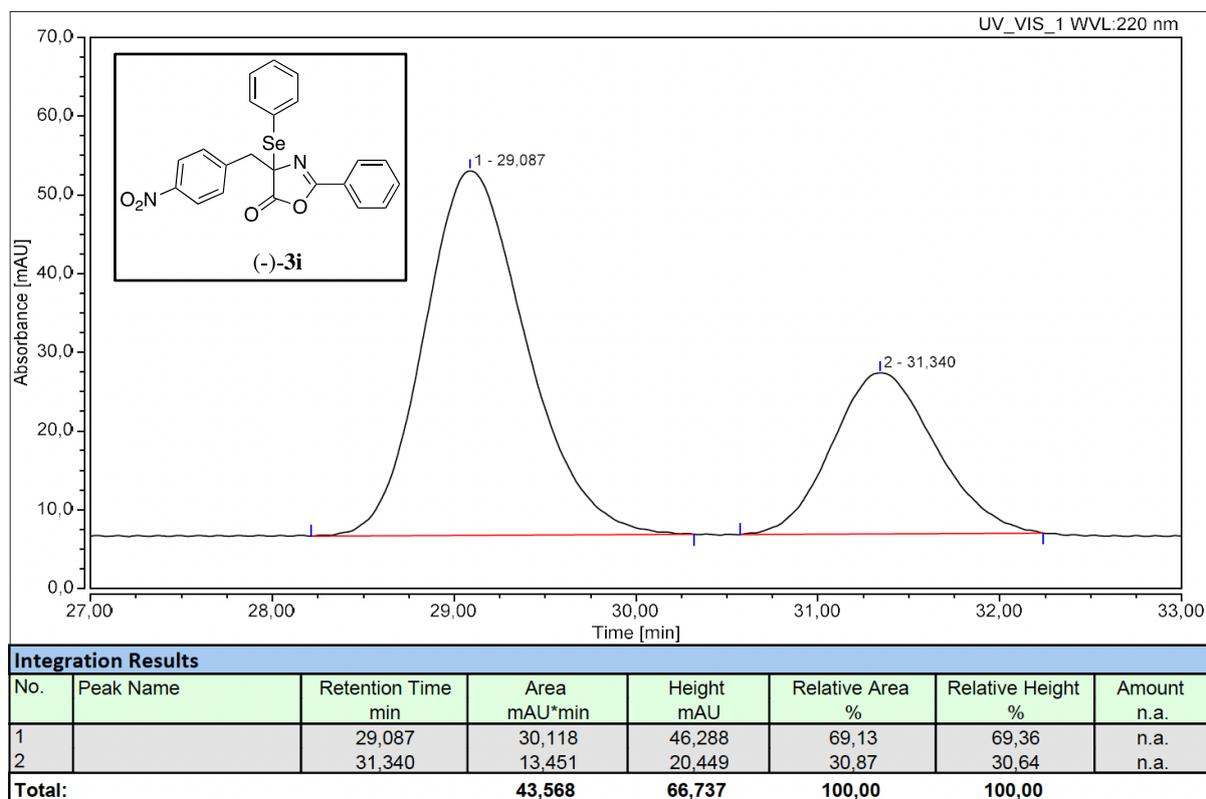
HPLC traces of **3g** (Chiralpak AD-H, *n*-hexane:*i*-PrOH = 20:1, flow rate 0.3 mL/min, 10 °C, $\lambda = 220$ nm)



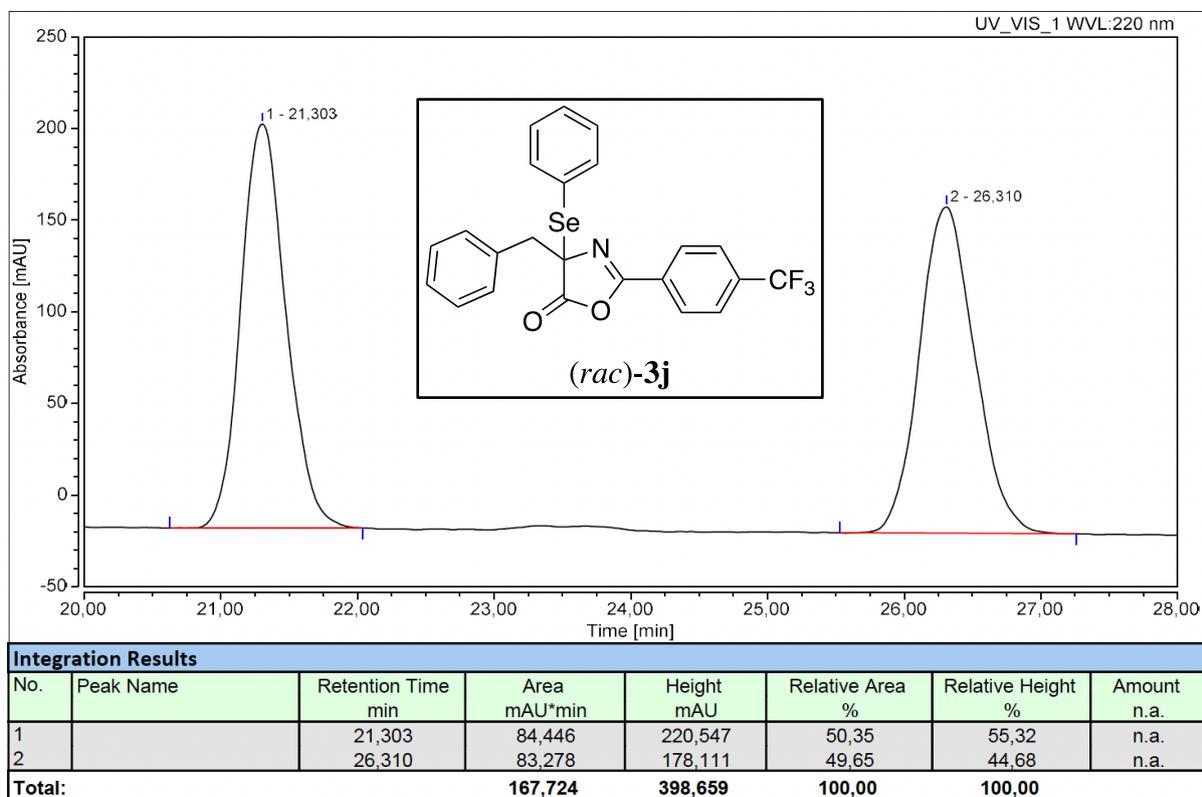
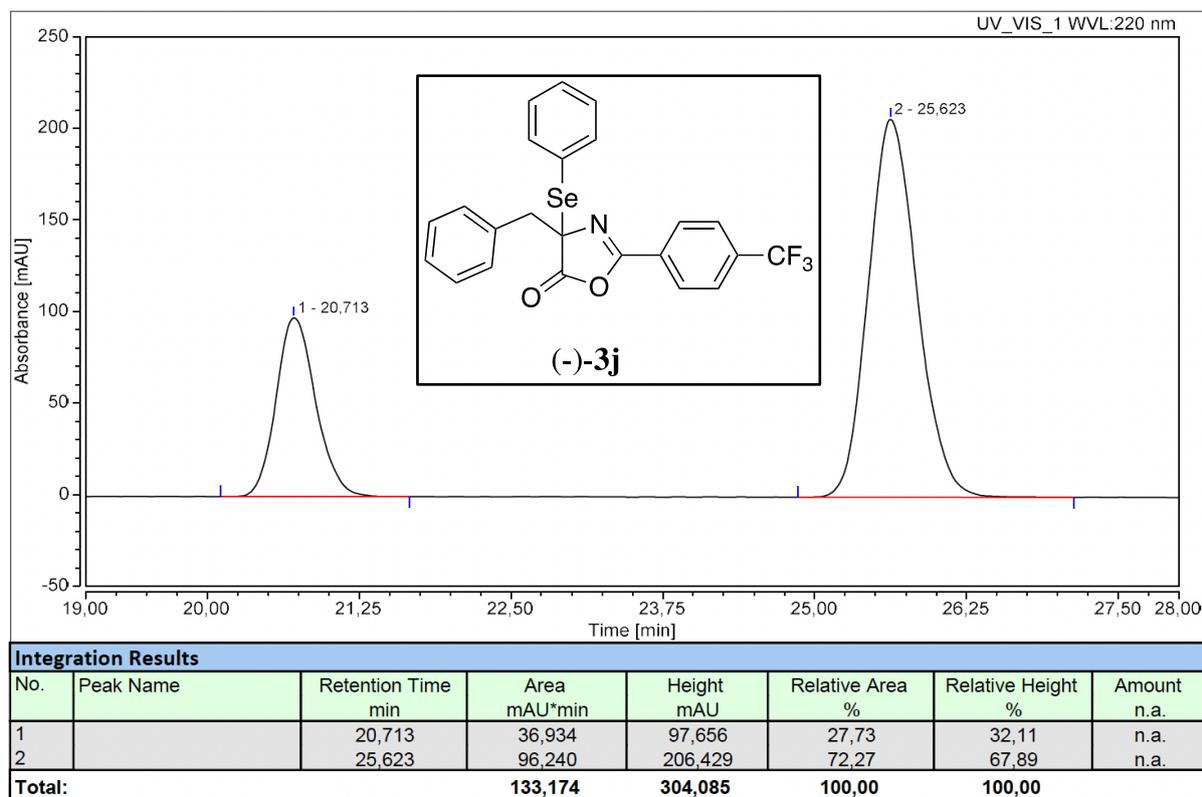
HPLC traces of **3h** (Chiralpak AD-H, *n*-hexane:*i*-PrOH = 20:1, flow rate 0.3 mL/min, 10 °C, $\lambda = 220$ nm)



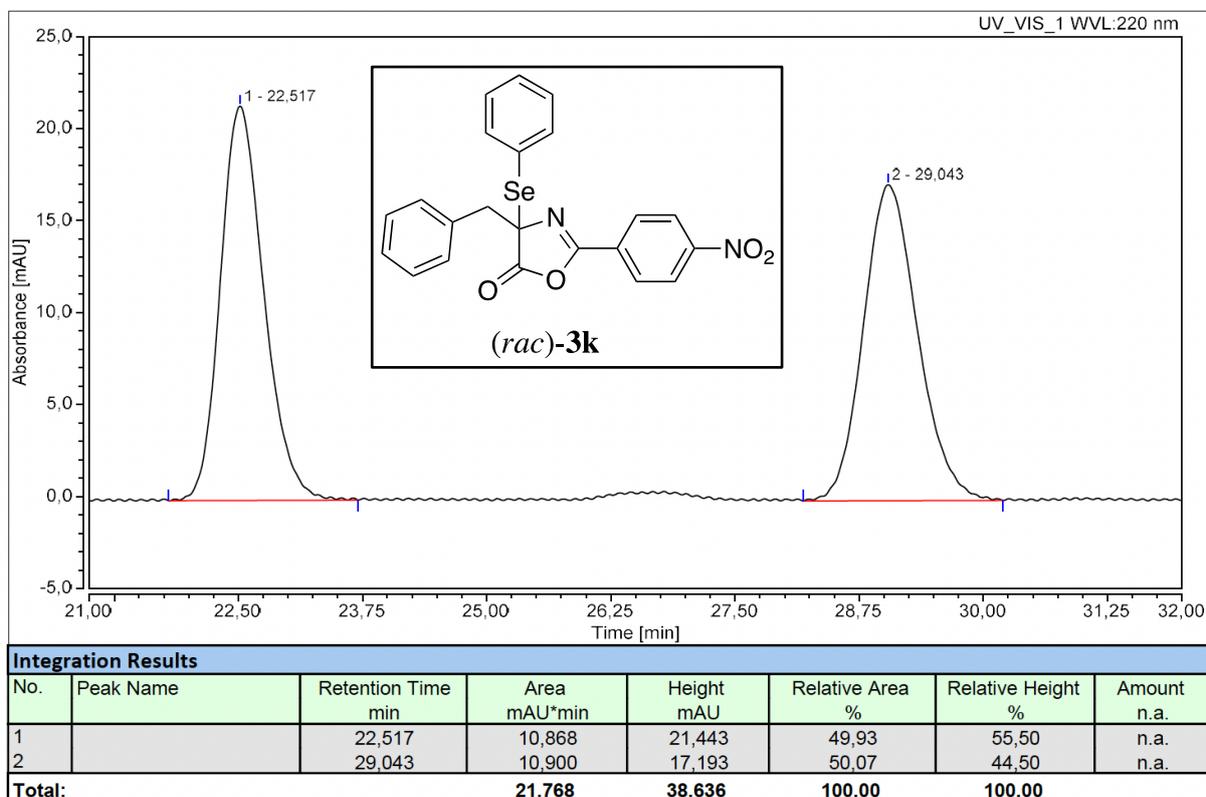
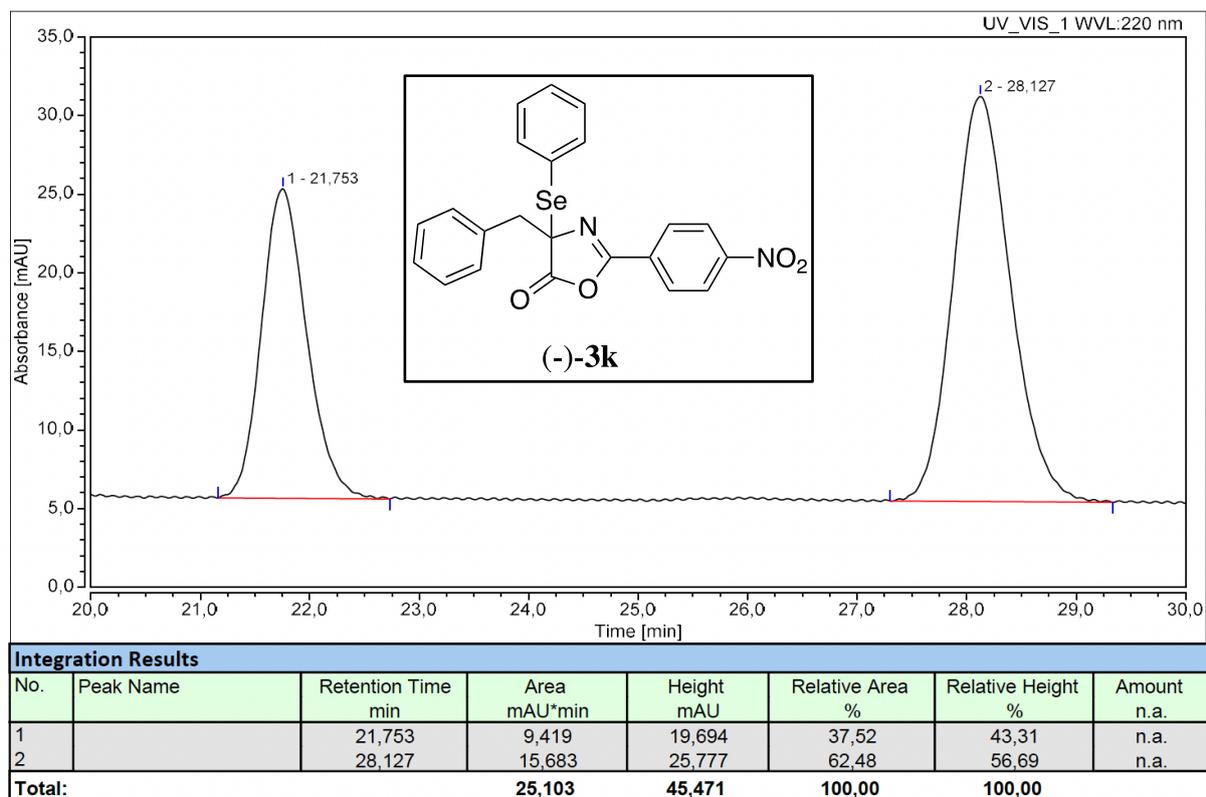
HPLC traces of **3i** (Chiralpak AD-H, *n*-hexane:*i*-PrOH = 20:1, flow rate 0.7 mL/min, 10 °C, $\lambda = 220$ nm)



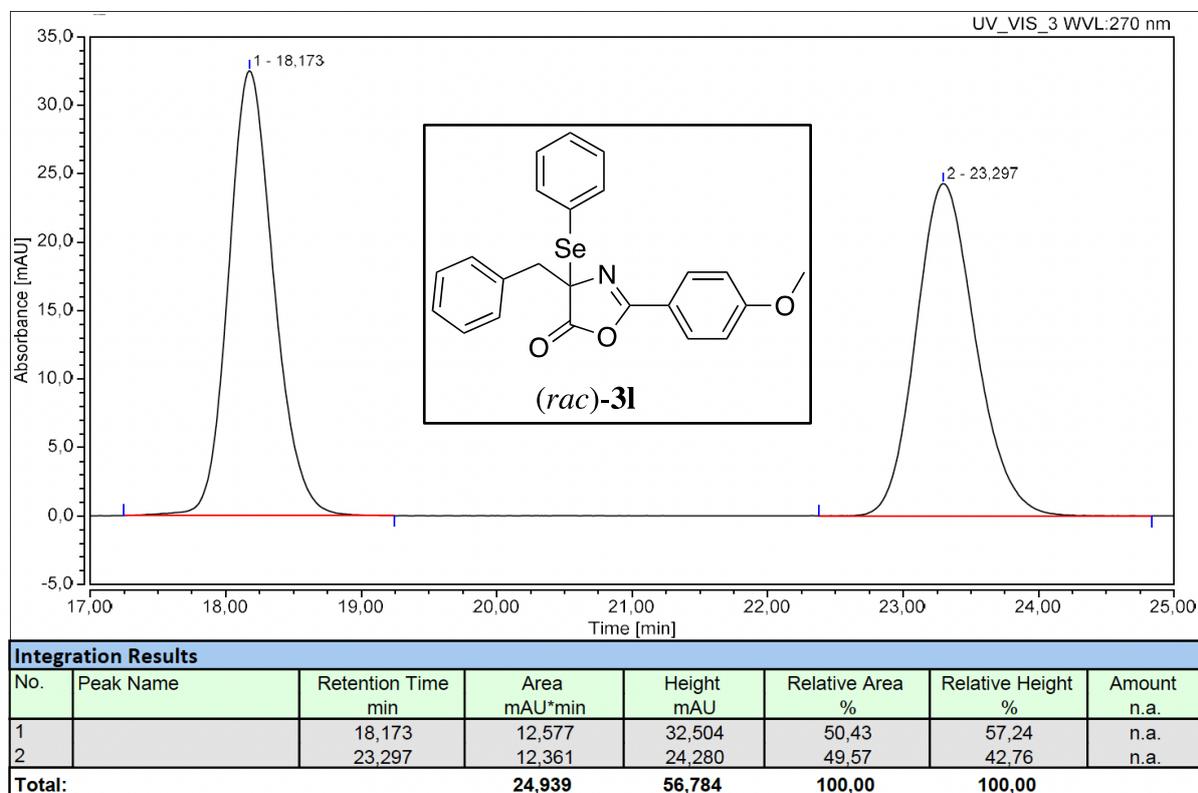
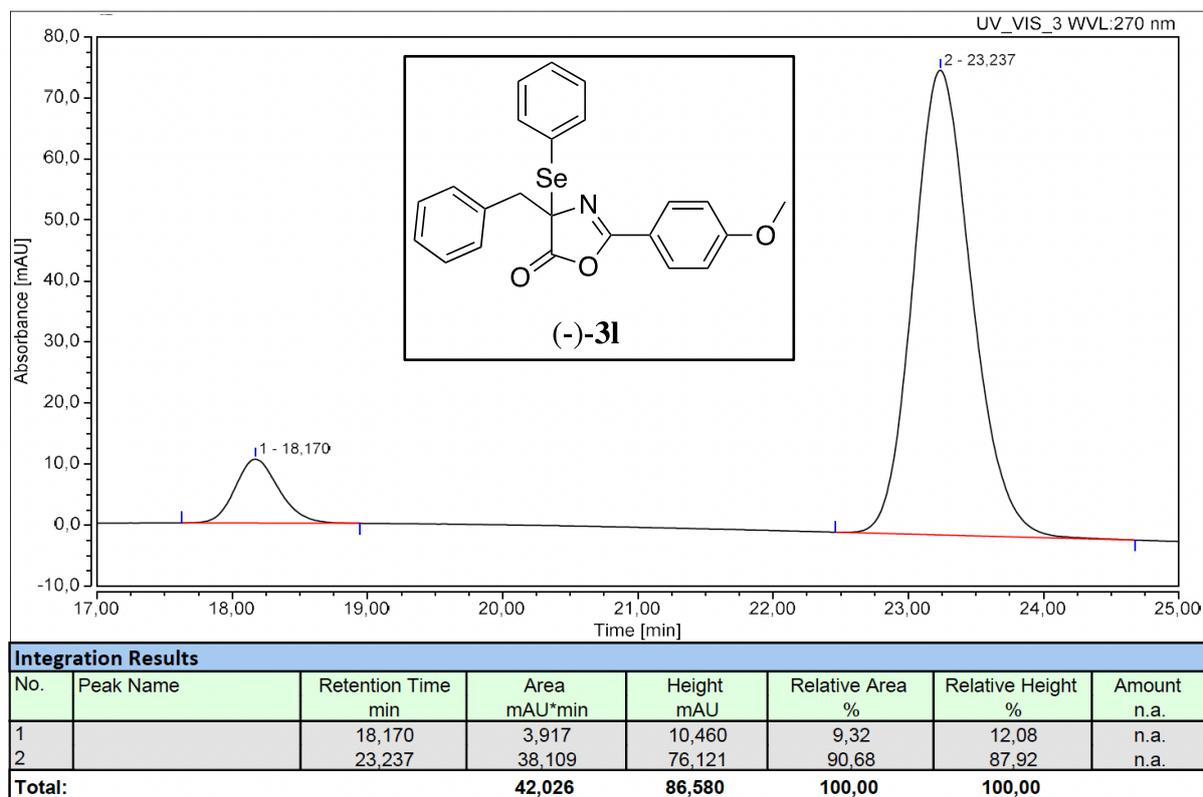
HPLC traces of **3j** (Chiralpak AD-H, *n*-hexane:*i*-PrOH = 20:1, flow rate 0.3 mL/min, 10 °C, $\lambda = 220$ nm)



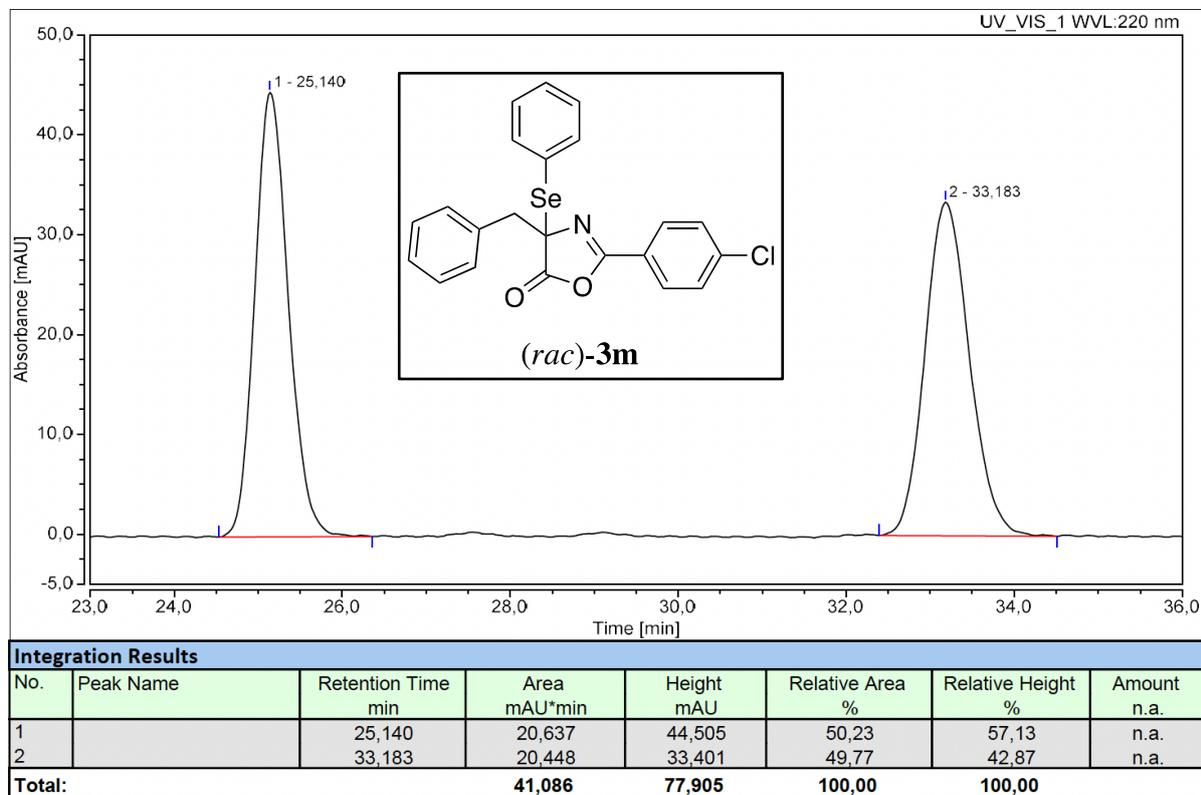
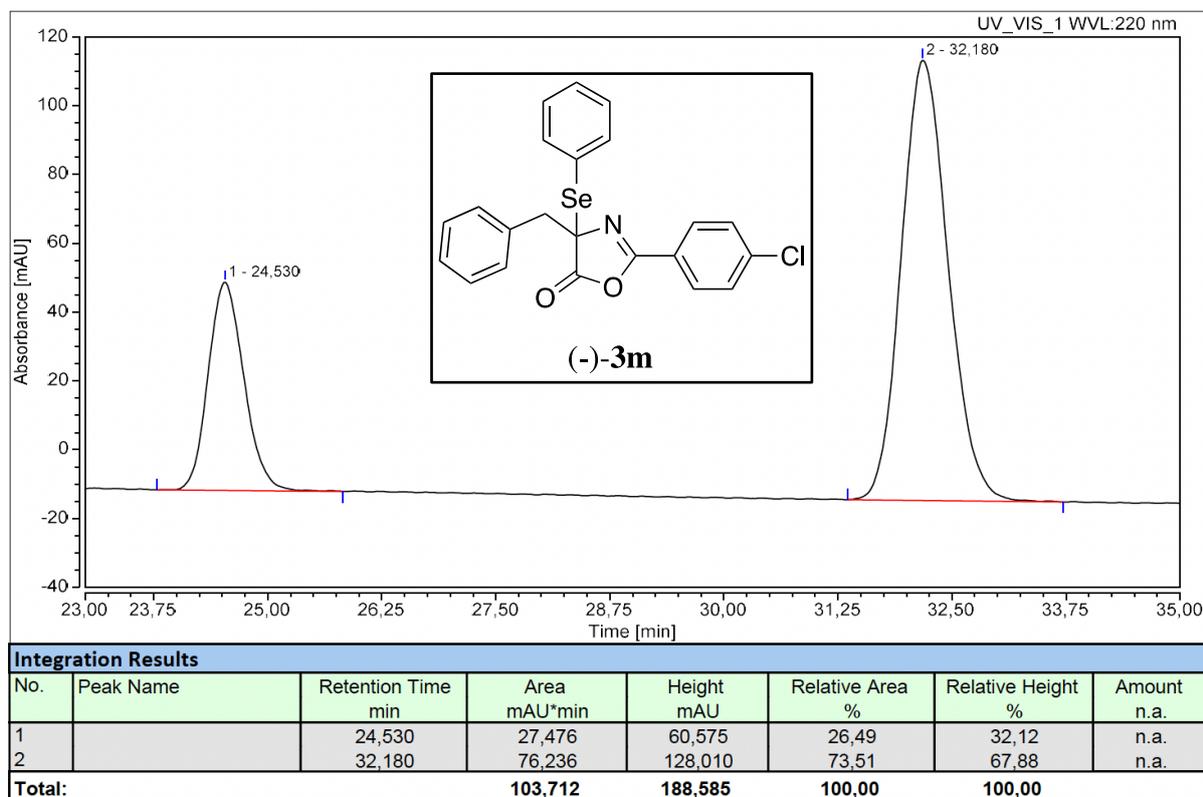
HPLC traces of **3k** (Chiralpak AD-H, *n*-hexane:*i*-PrOH = 20:1, flow rate 0.7 mL/min, 10 °C, $\lambda = 220$ nm)



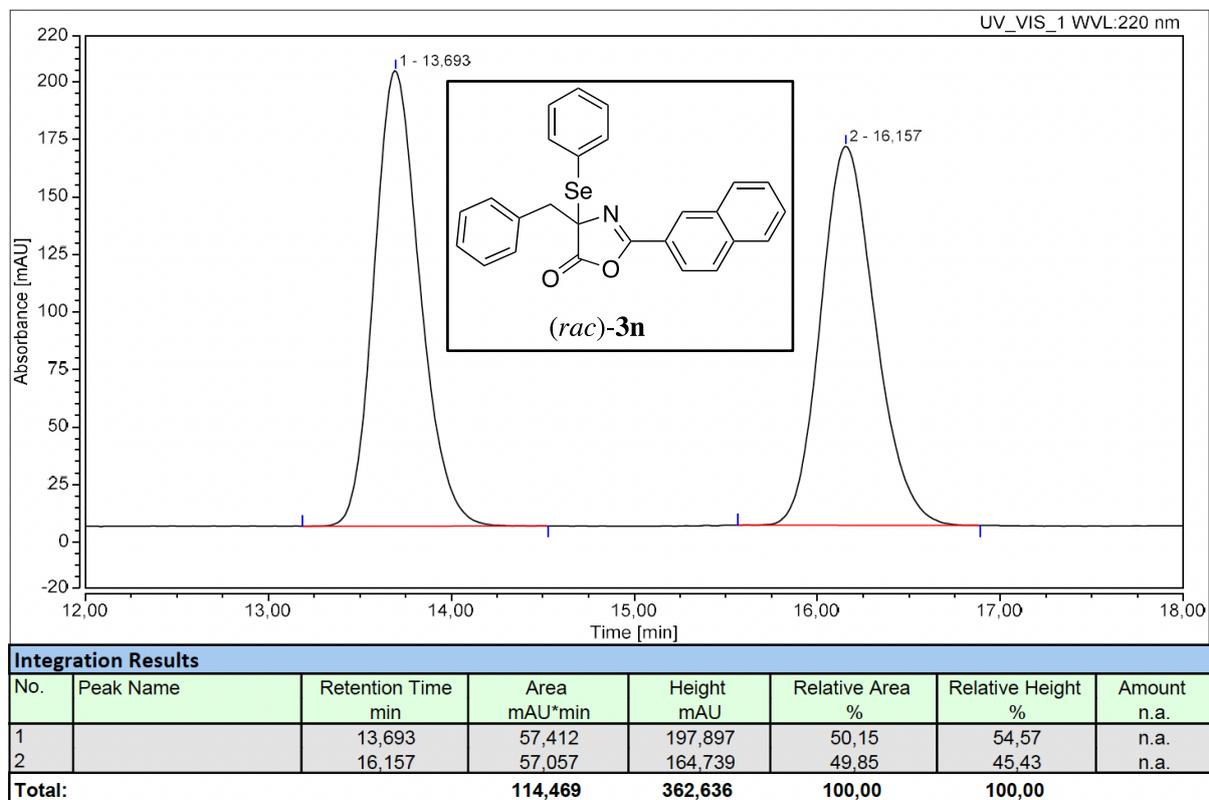
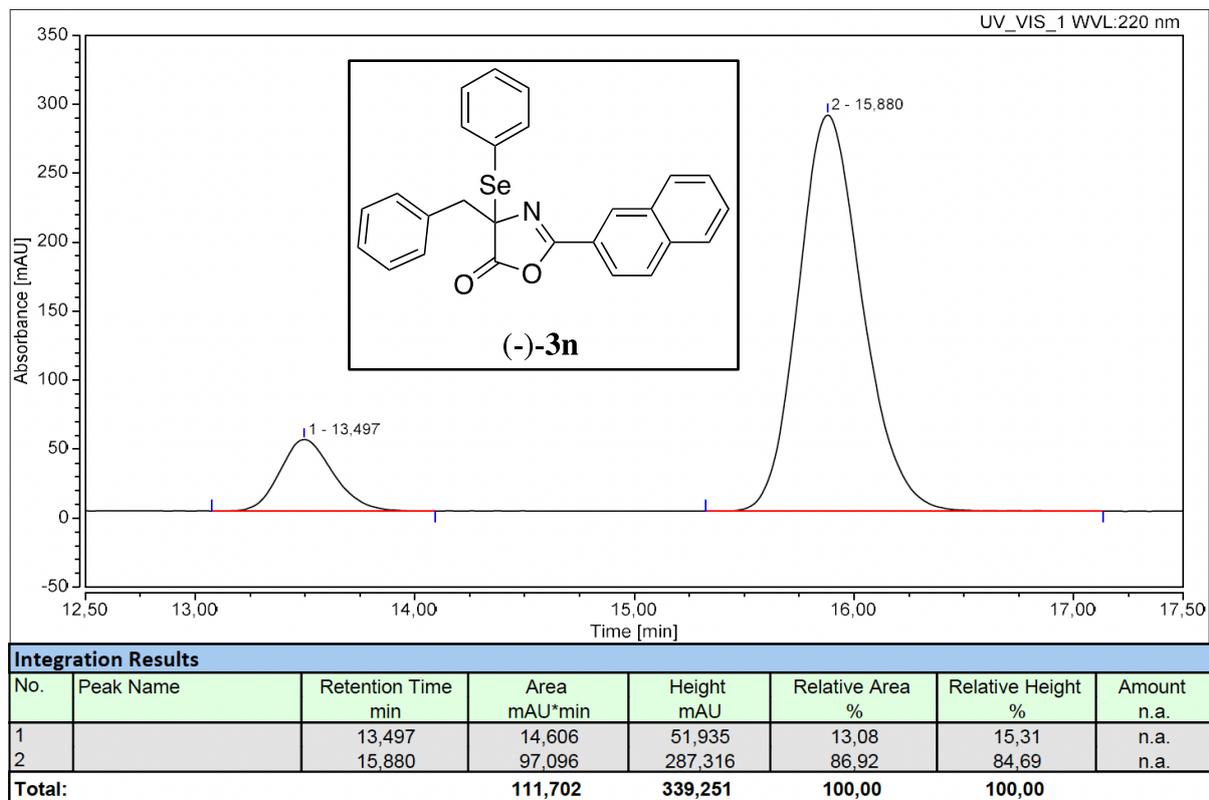
HPLC traces of **31** (Chiralpak AD-H, *n*-hexane:*i*-PrOH = 20:1, flow rate 0.7 mL/min, 10 °C, $\lambda = 270$ nm)



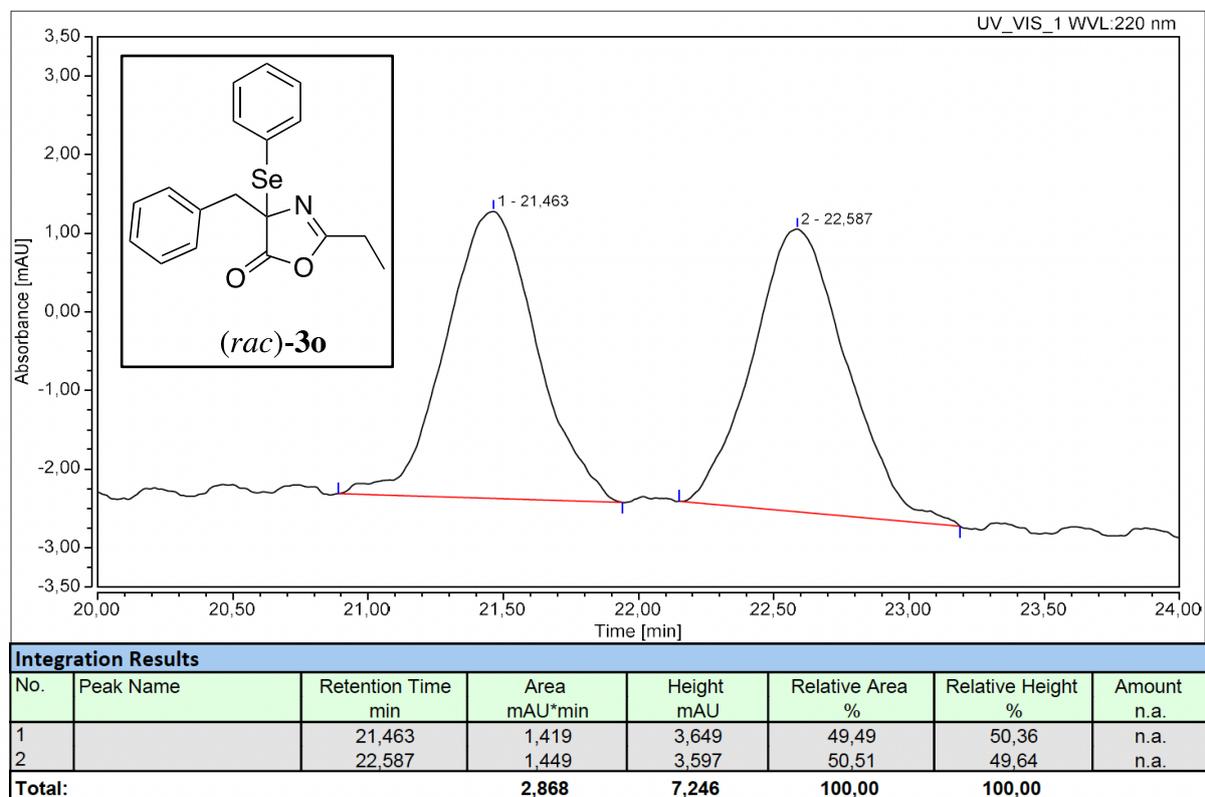
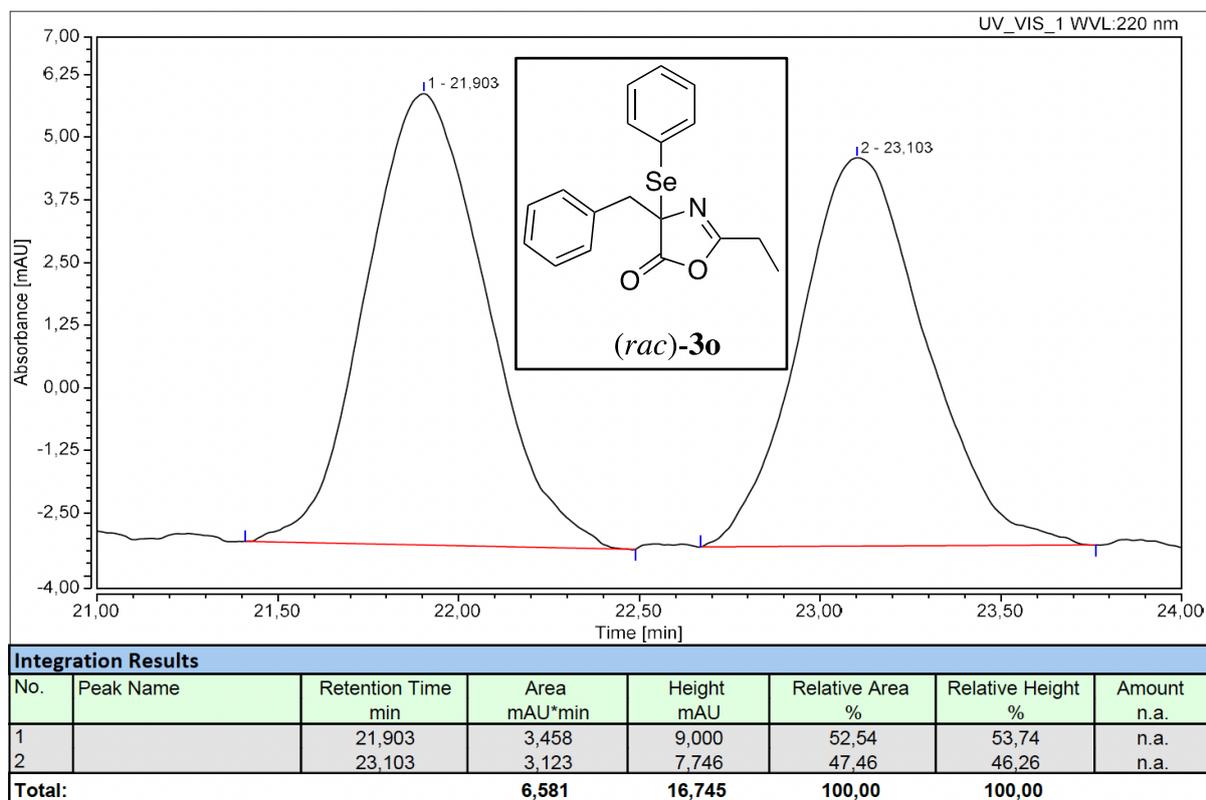
HPLC traces of **3m** (Chiralpak AD-H, *n*-hexane:*i*-PrOH = 20:1, flow rate 0.3 mL/min, 10 °C, λ = 220 nm)



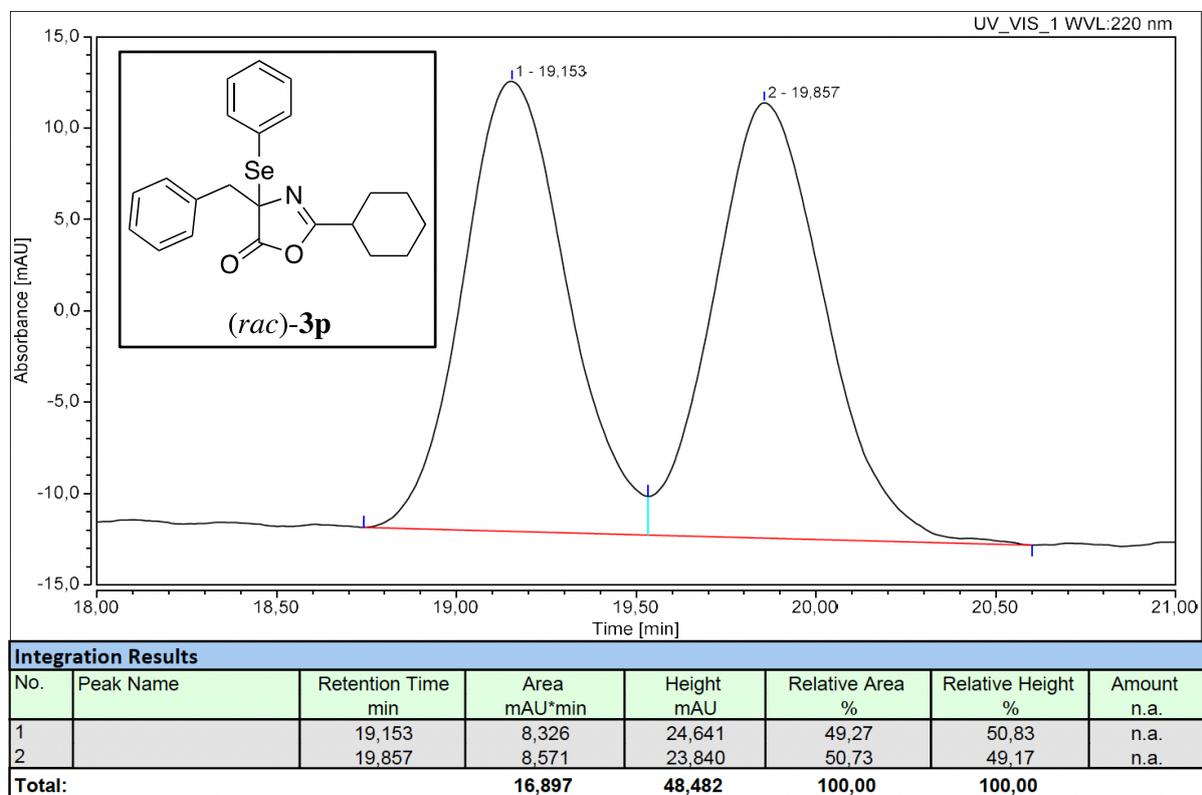
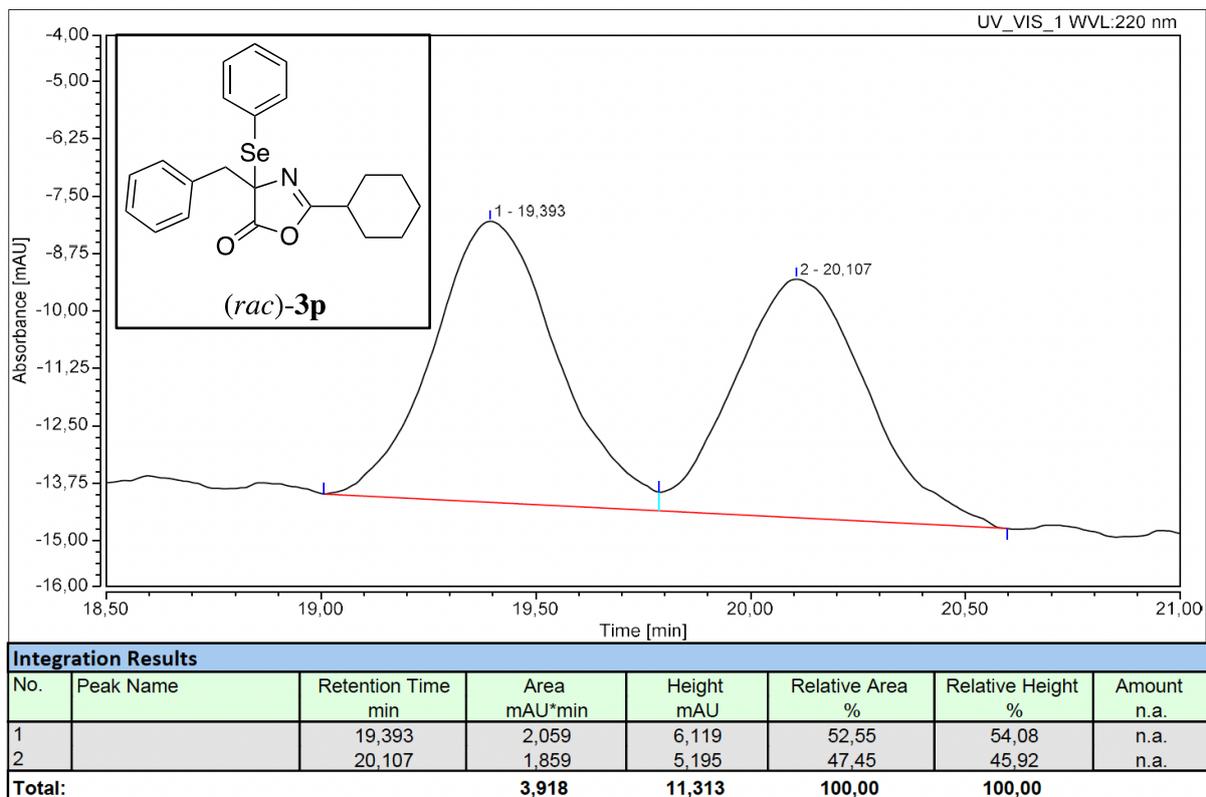
HPLC traces of **3n** (Chiralpak AD-H, *n*-hexane:*i*-PrOH = 20:1, flow rate 0.7 mL/min, 10 °C, $\lambda = 220$ nm)



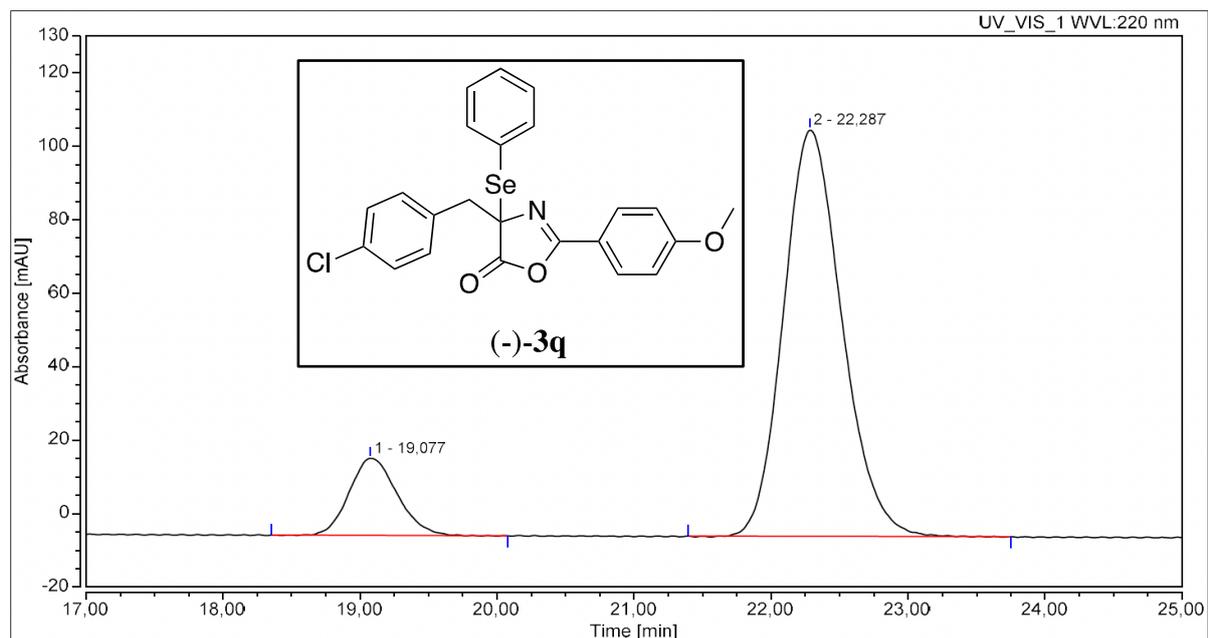
HPLC traces of **3o** (Chiralpak AD-H, *n*-hexane:*i*-PrOH = 20:1, flow rate 0.3 mL/min, 10 °C, $\lambda = 220$ nm)



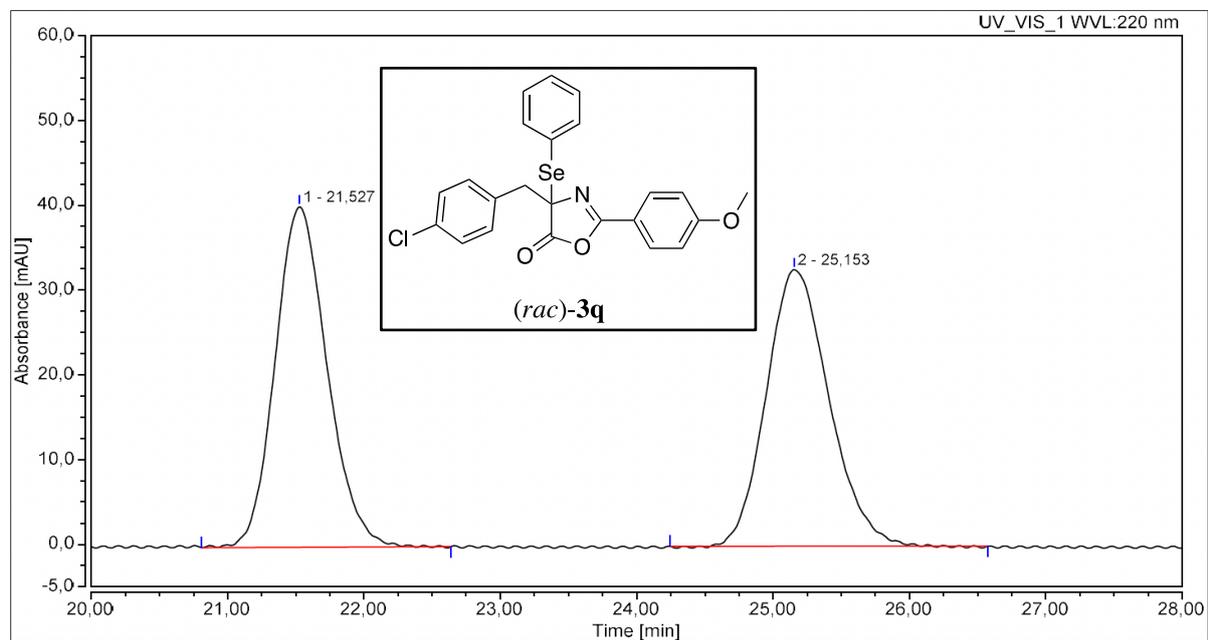
HPLC traces of **3p** (Chiralpak AD-H, *n*-hexane:*i*-PrOH = 20:1, flow rate 0.3 mL/min, 10 °C, $\lambda = 220$ nm)



HPLC traces of **3q** (Chiralpak AD-H, *n*-hexane:*i*-PrOH = 20:1, flow rate 0.7 mL/min, 10 °C, $\lambda = 220$ nm)

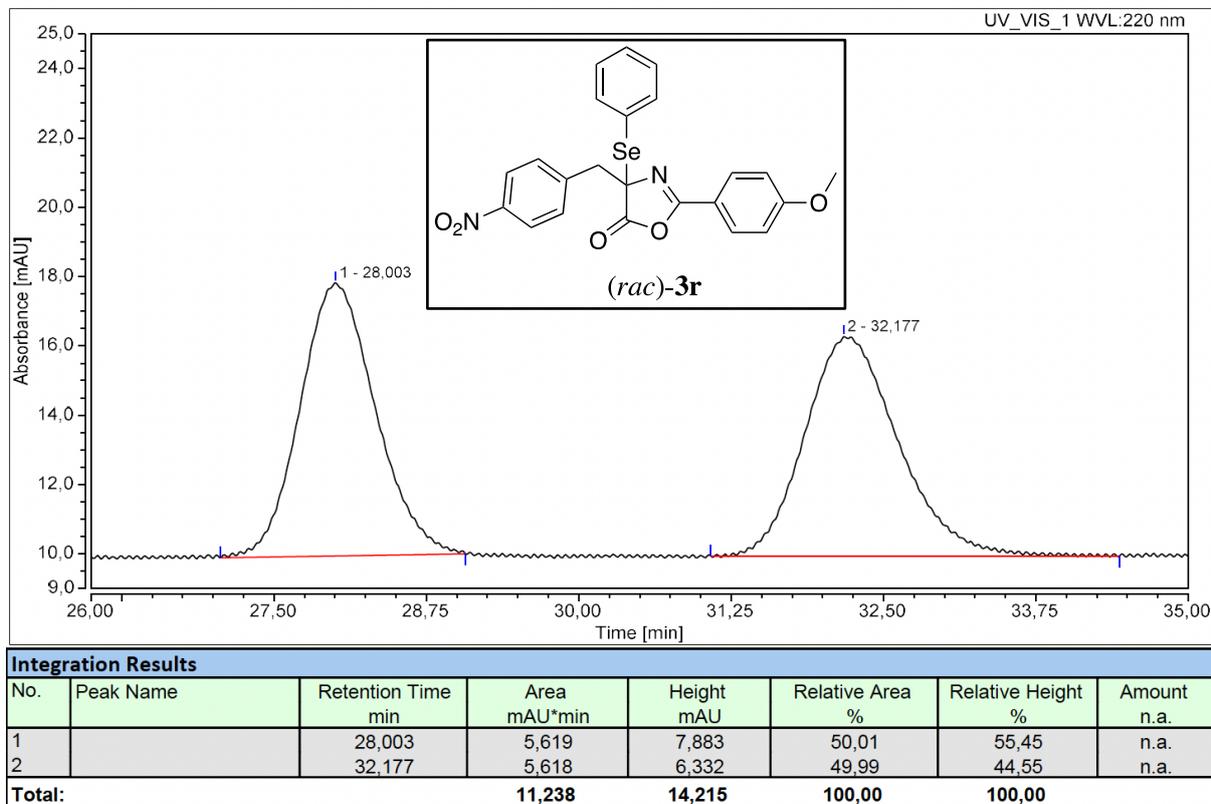
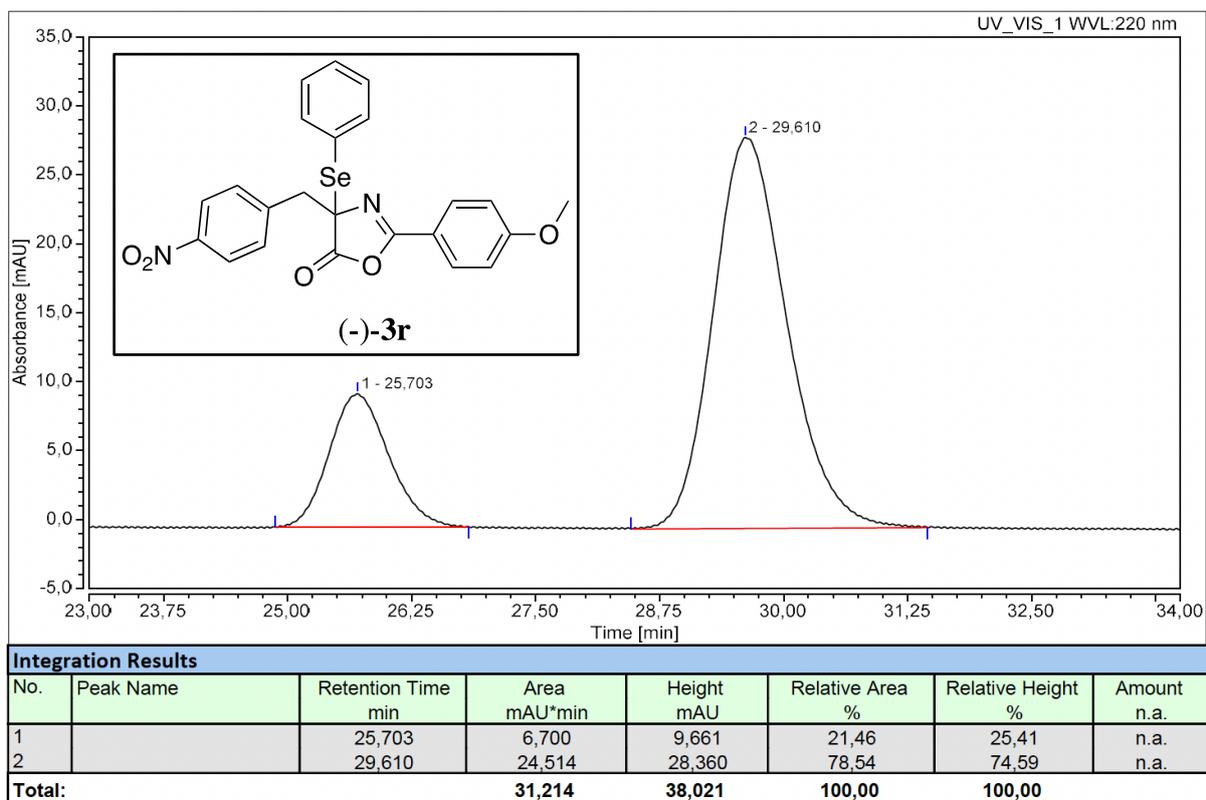


Integration Results							
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		19,077	8,522	20,974	13,50	15,94	n.a.
2		22,287	54,584	110,590	86,50	84,06	n.a.
Total:			63,106	131,565	100,00	100,00	

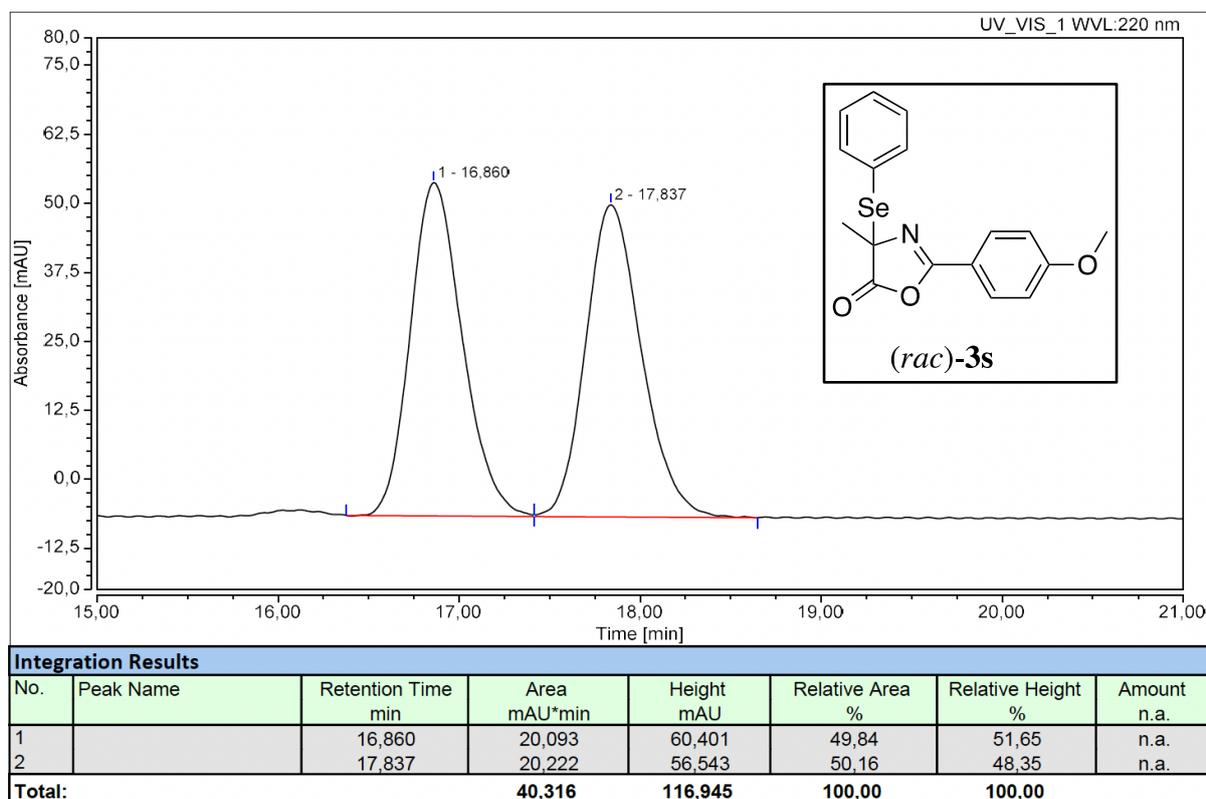
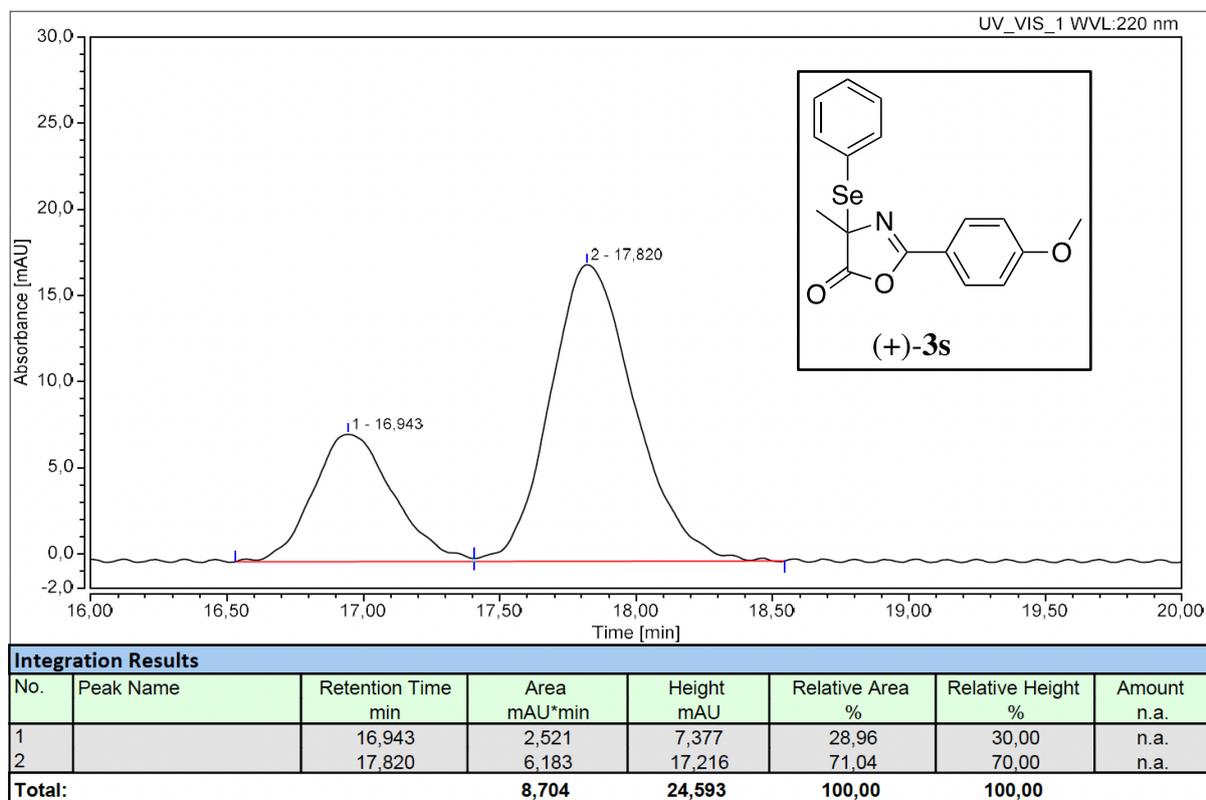


Integration Results							
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		21,527	17,653	40,156	50,48	55,17	n.a.
2		25,153	17,318	32,632	49,52	44,83	n.a.
Total:			34,971	72,787	100,00	100,00	

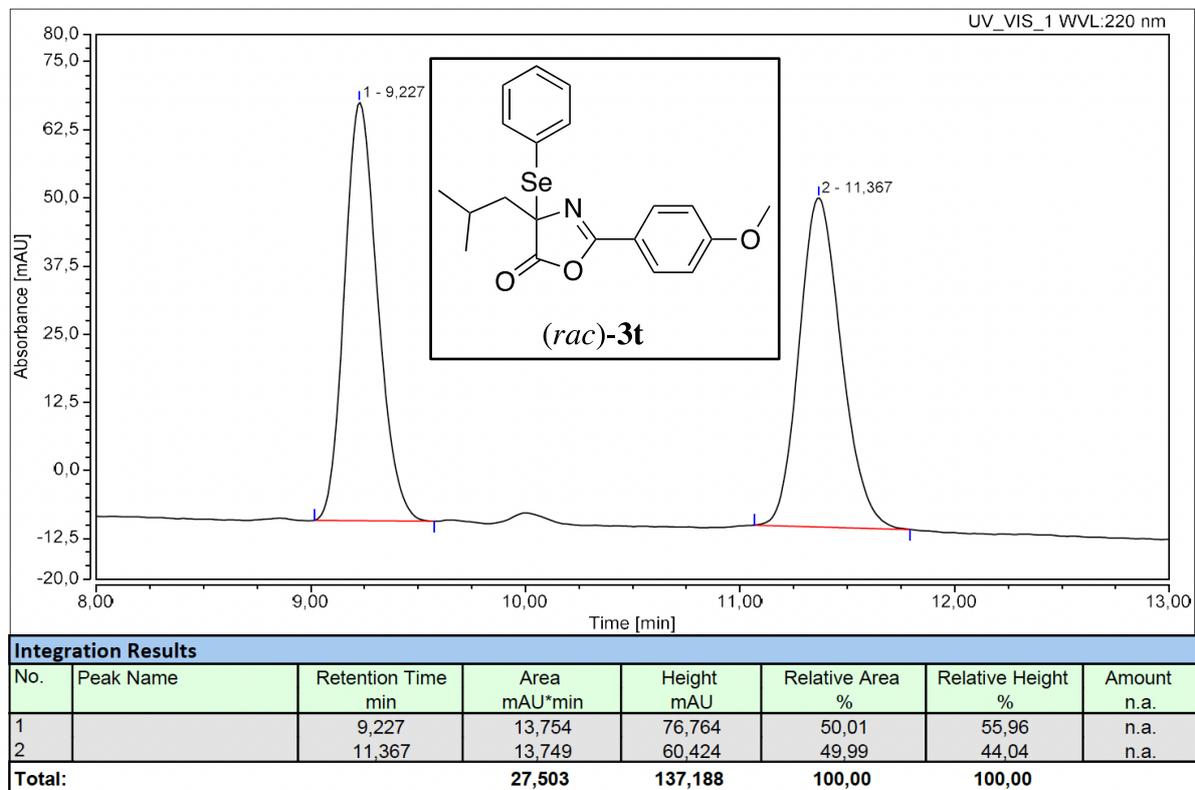
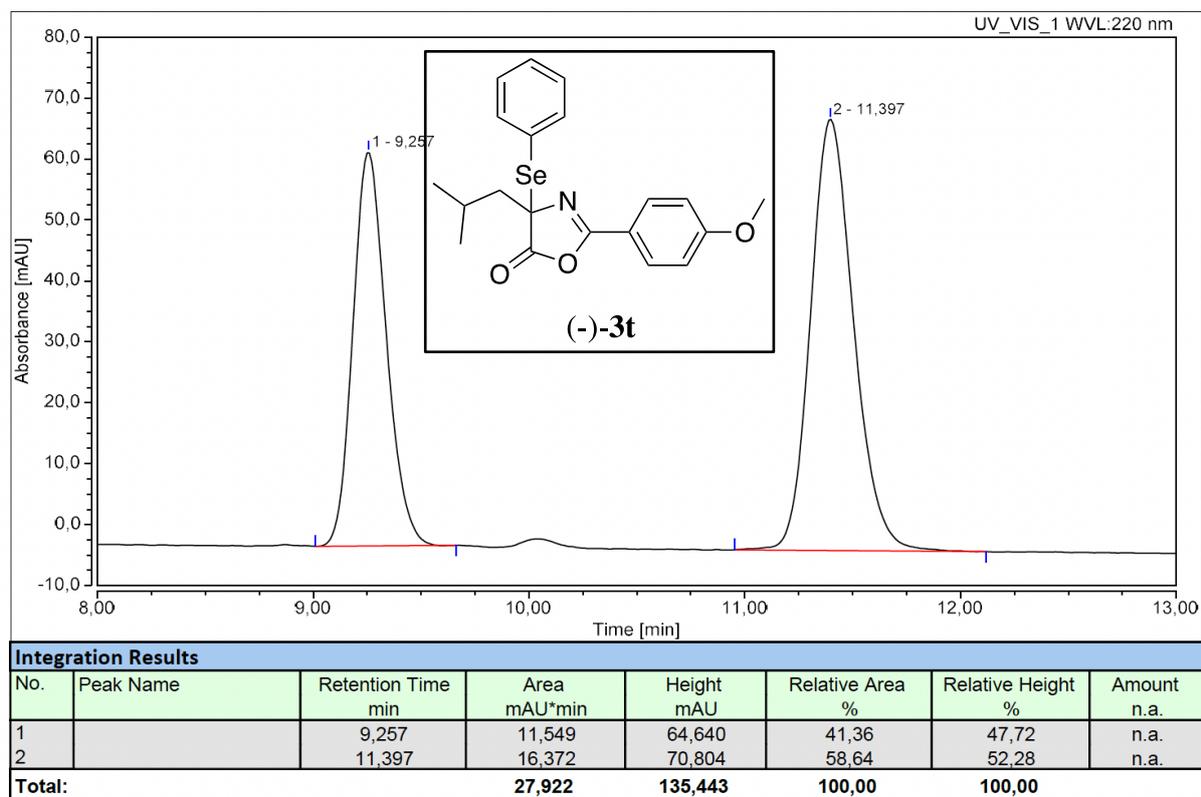
HPLC traces of **3r** (Chiralpak AD-H, *n*-hexane:*i*-PrOH = 20:1, flow rate 1.2 mL/min, 10 °C, $\lambda = 220$ nm)



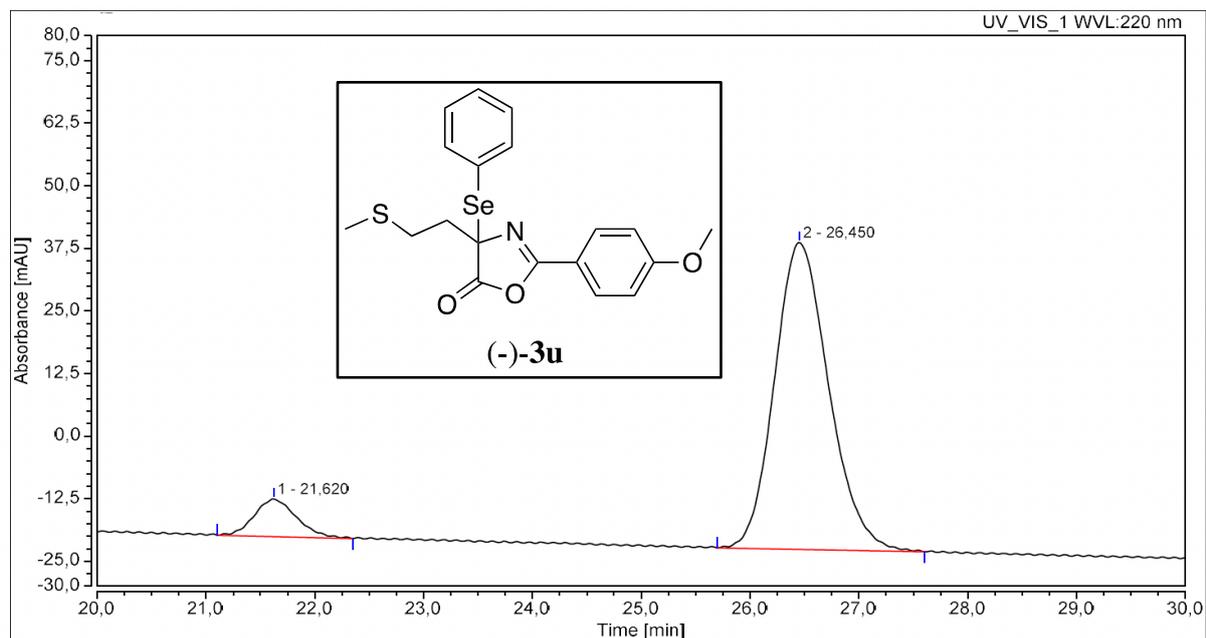
HPLC traces of 3s (Chiralpak AD-H, *n*-hexane:*i*-PrOH = 20:1, flow rate 0.7 mL/min, 10 °C, $\lambda = 220$ nm)



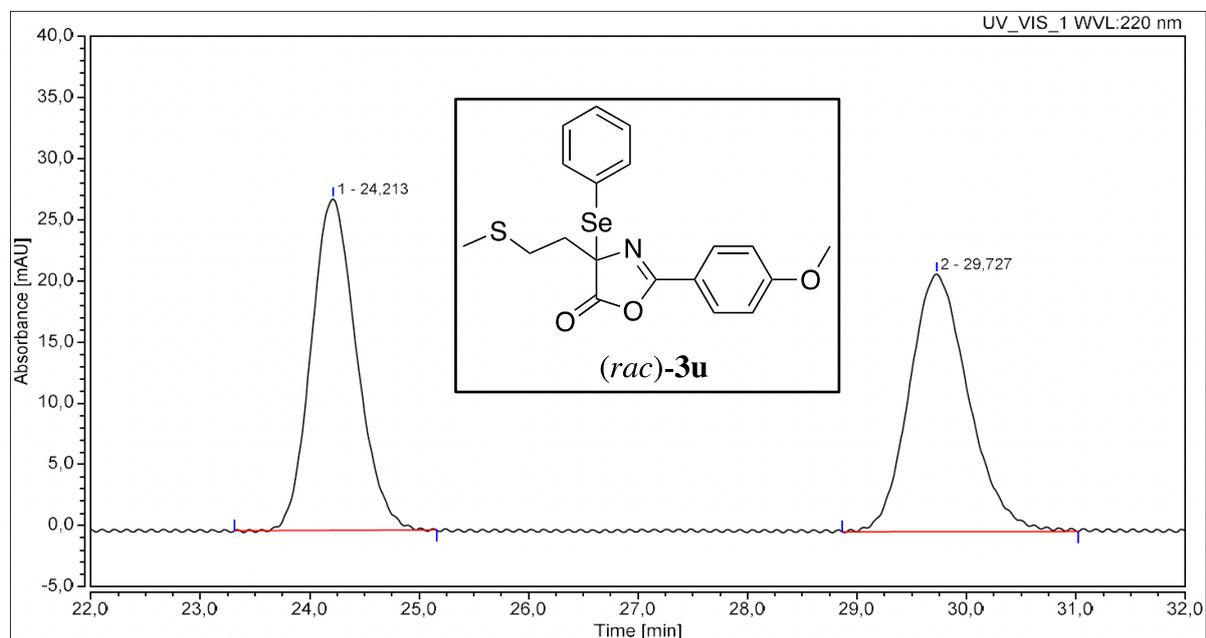
HPLC traces of **3t** (Chiralpak AD-H, *n*-hexane:*i*-PrOH = 20:1, flow rate 0.7 mL/min, 10 °C, $\lambda = 220$ nm)



HPLC traces of **3u** (Chiralpak AD-H, *n*-hexane:*i*-PrOH = 20:1, flow rate 0.7 mL/min, 10 °C, $\lambda = 220$ nm)

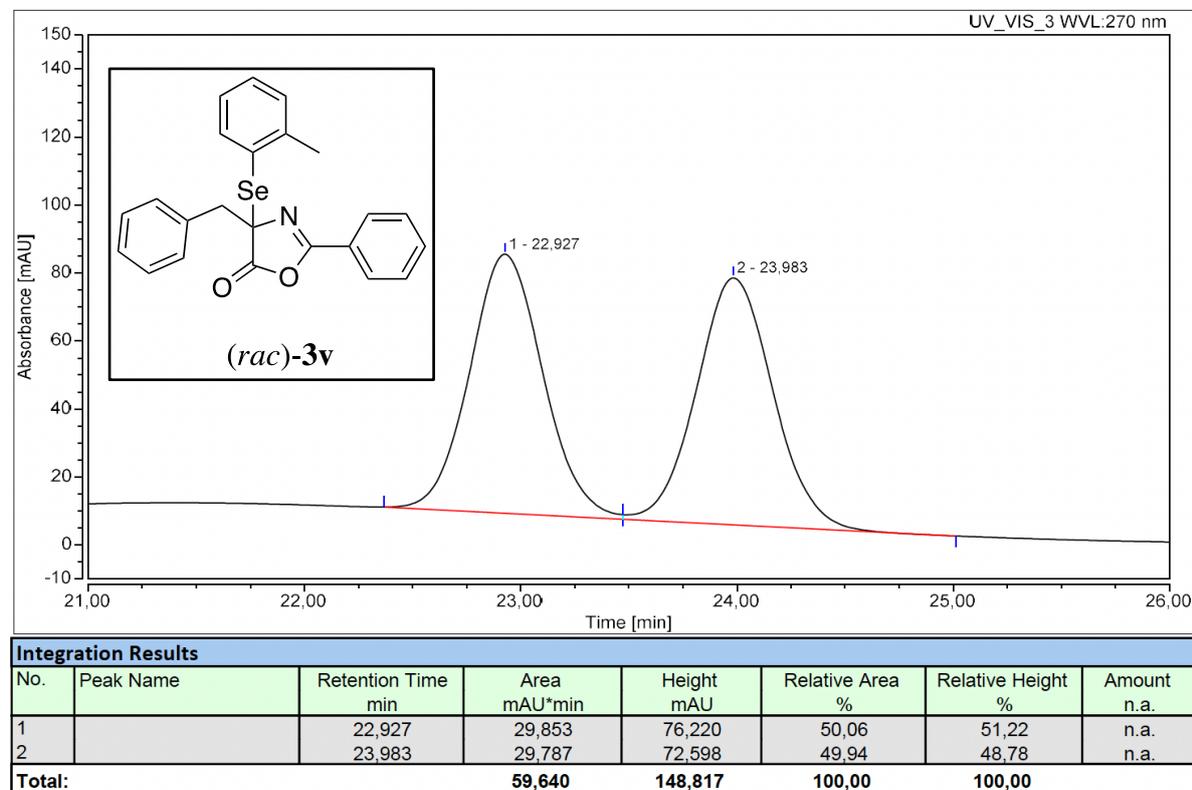
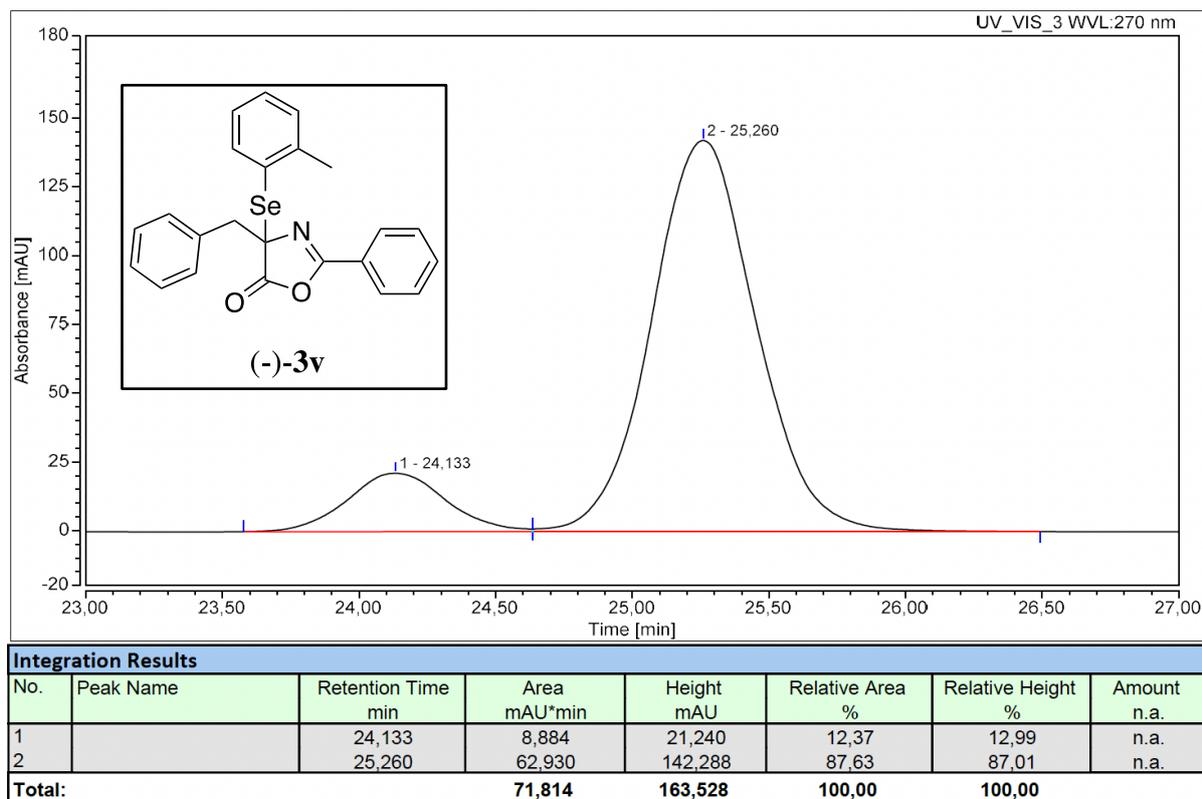


Integration Results							
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		21,620	3,374	7,523	8,70	10,93	n.a.
2		26,450	35,418	61,311	91,30	89,07	n.a.
Total:			38,792	68,834	100,00	100,00	

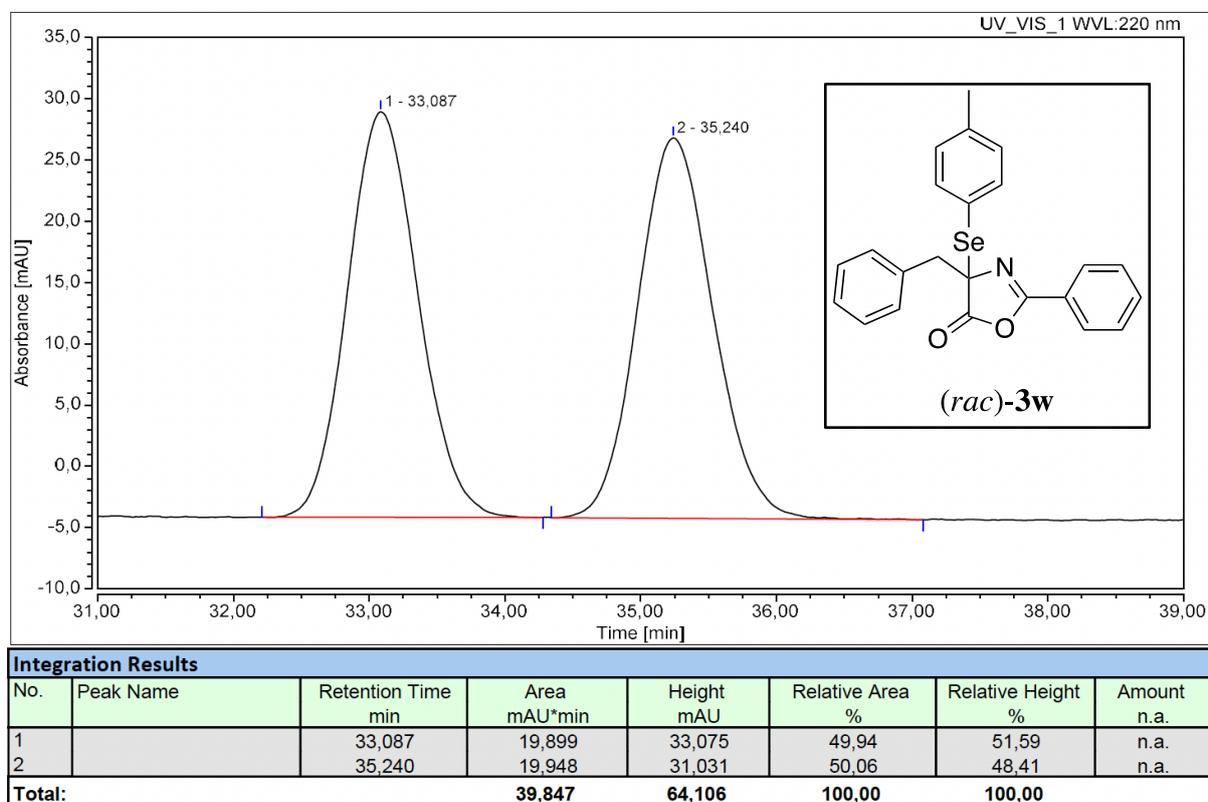
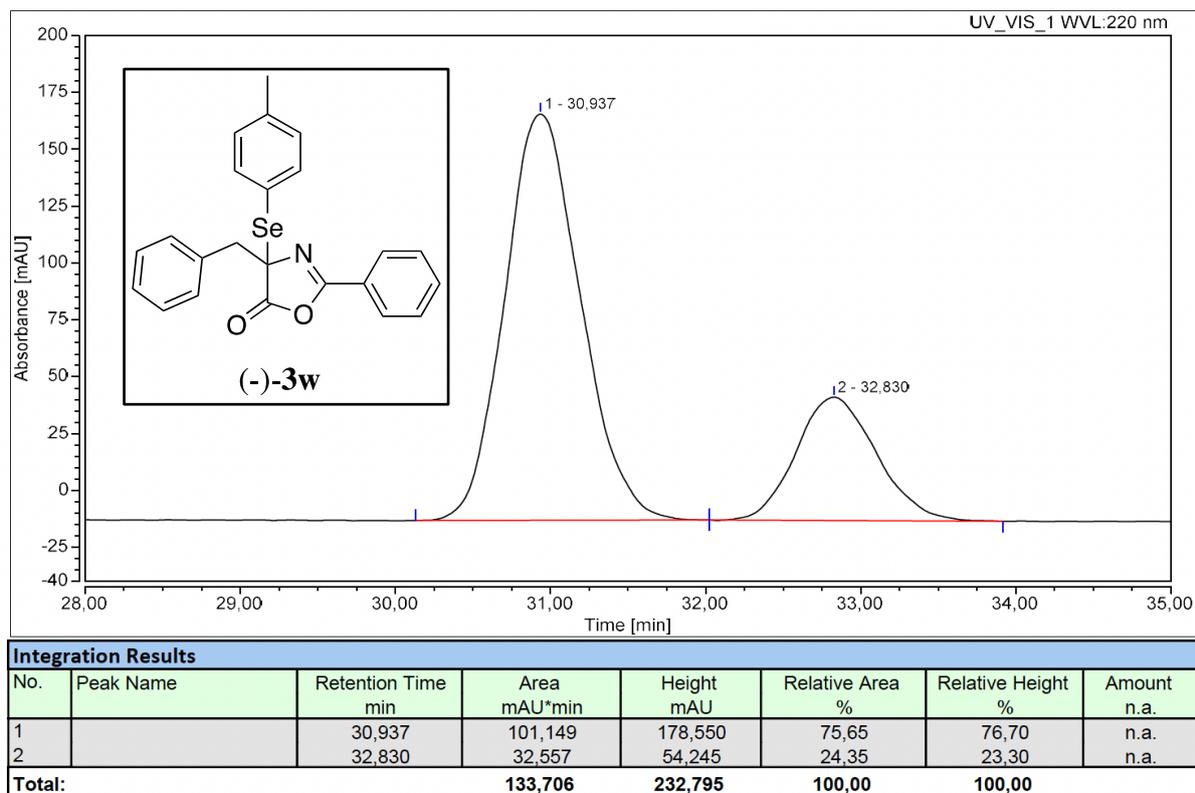


Integration Results							
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		24,213	13,040	27,096	49,74	56,27	n.a.
2		29,727	13,175	21,057	50,26	43,73	n.a.
Total:			26,214	48,153	100,00	100,00	

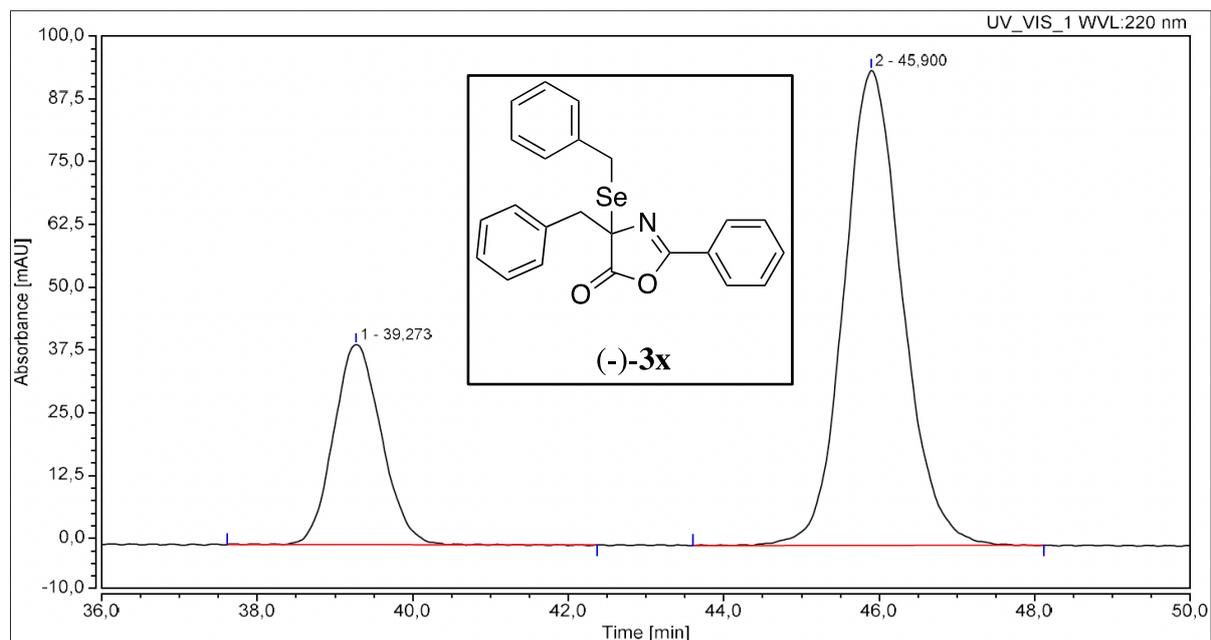
HPLC traces of 3v (YMC Chiral Art Cellulose SB, *n*-hexane:*i*-PrOH = 20:1, flow rate 0.3 mL/min, 10 °C, λ = 270 nm)



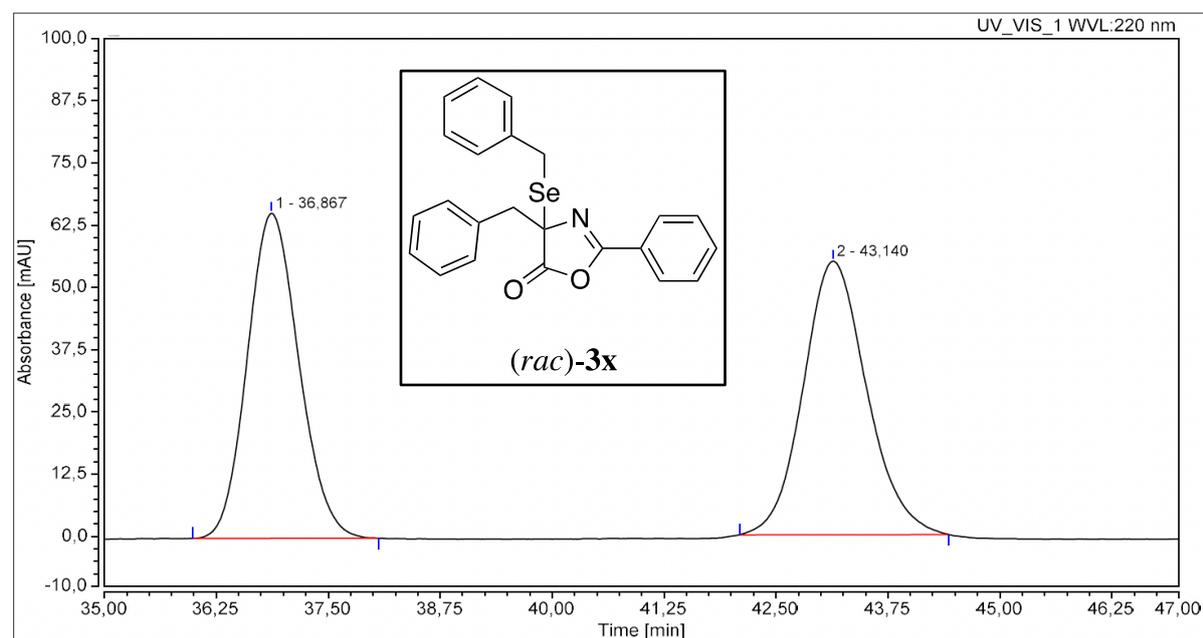
HPLC traces of **3w** (Chiralpak AD-H, *n*-hexane:*i*-PrOH = 20:1, flow rate 0.3 mL/min, 10 °C, $\lambda = 220$ nm)



HPLC traces of **3x** (Chiralpak AD-H, *n*-hexane:*i*-PrOH = 20:1, flow rate 0.3 mL/min, 10 °C, $\lambda = 220$ nm)

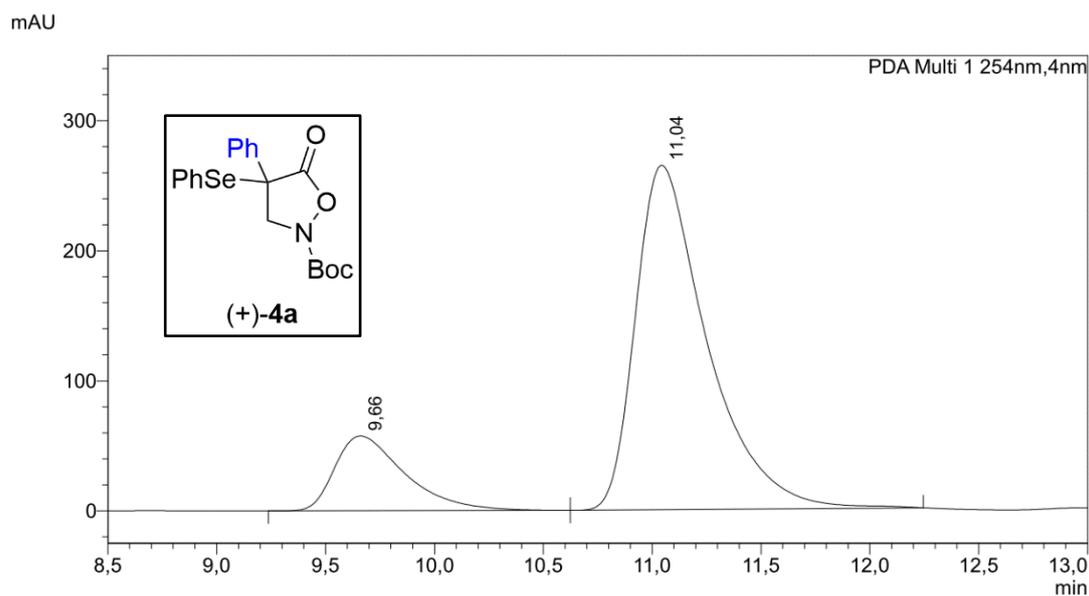


Integration Results							
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		39,273	28,368	39,823	25,71	29,63	n.a.
2		45,900	81,976	94,563	74,29	70,37	n.a.
Total:			110,344	134,385	100,00	100,00	



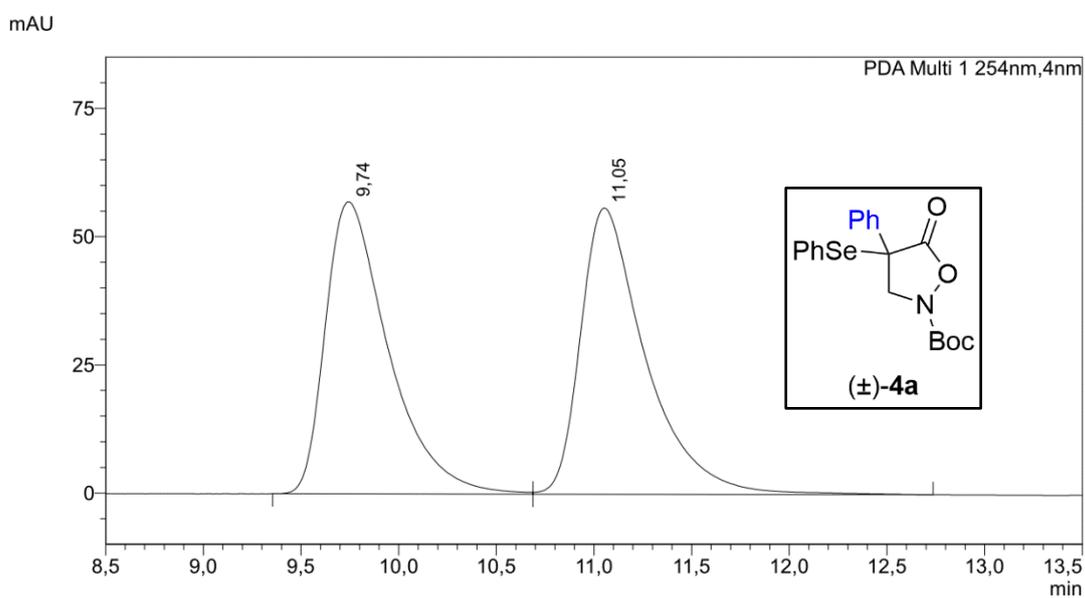
Integration Results							
No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount n.a.
1		36,867	43,568	65,307	49,14	54,31	n.a.
2		43,140	45,088	54,939	50,86	45,69	n.a.
Total:			88,657	120,245	100,00	100,00	

HPLC traces of **4a** (Chiralpak AD-H, *n*-hexane:*i*-PrOH = 20:1, flow rate 1.0 mL/min, 20 °C, $\lambda = 254$ nm)



Peak Table

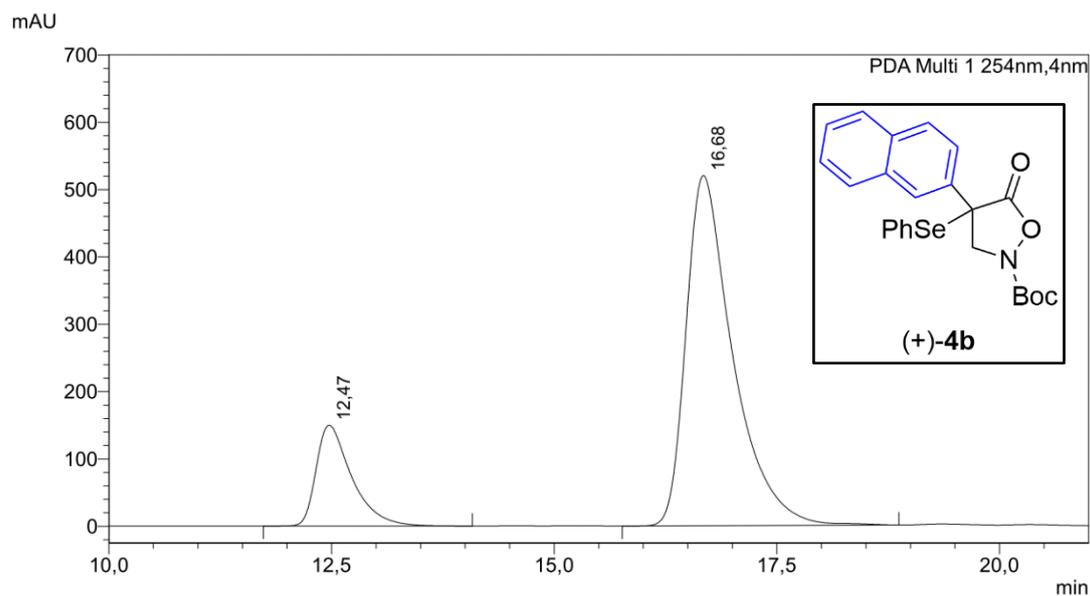
Peak#	Ret. Time	Area	Area%
1	9,66	1282010	17,45
2	11,04	6065478	82,55
Total		7347487	100,00



Peak Table

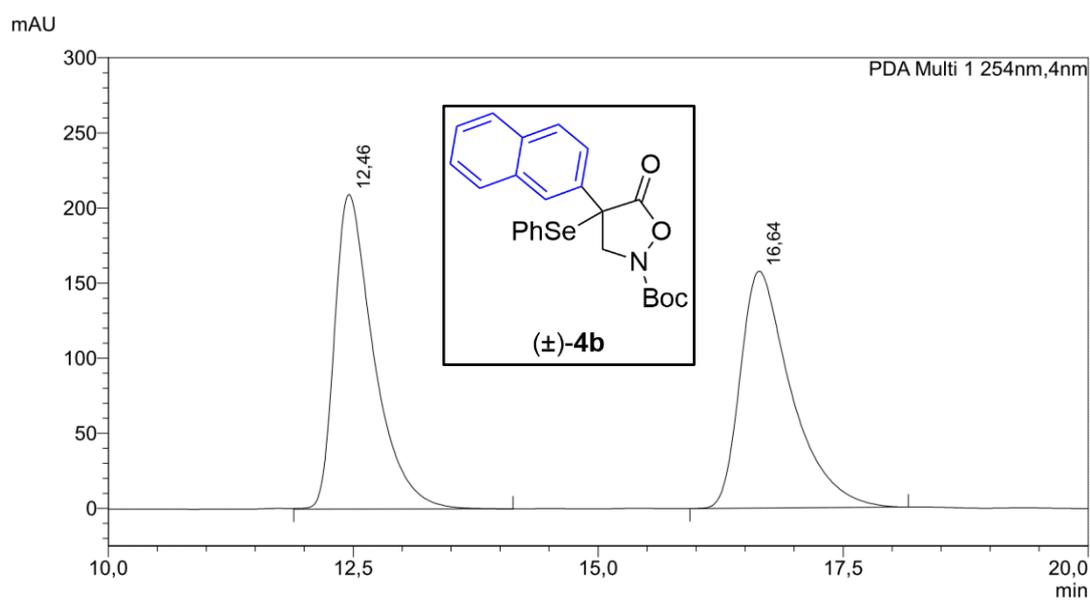
Peak#	Ret. Time	Area	Area%
1	9,74	1277340	49,66
2	11,05	1294957	50,34
Total		2572297	100,00

HPLC traces of **4b** (Chiralpak AD-H, *n*-hexane:*i*-PrOH = 20:1, flow rate 1.0 mL/min, 20 °C, $\lambda = 254$ nm)



Peak Table

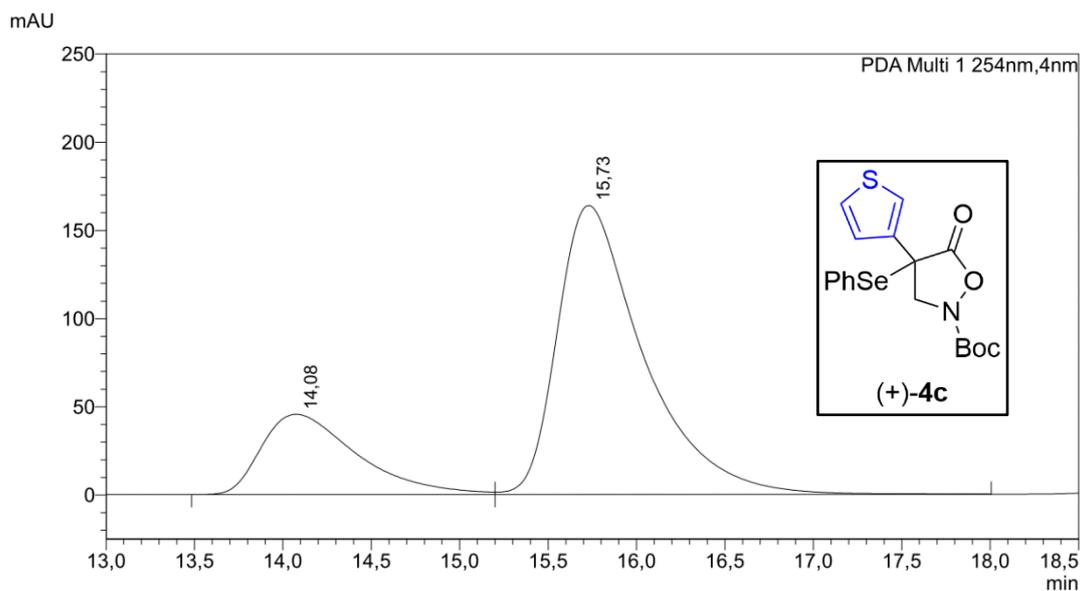
Peak#	Ret. Time	Area	Area%
1	12,47	4169553	17,97
2	16,68	19033478	82,03
Total		23203031	100,00



Peak Table

Peak#	Ret. Time	Area	Area%
1	12,46	5822250	50,46
2	16,64	5715966	49,54
Total		11538216	100,00

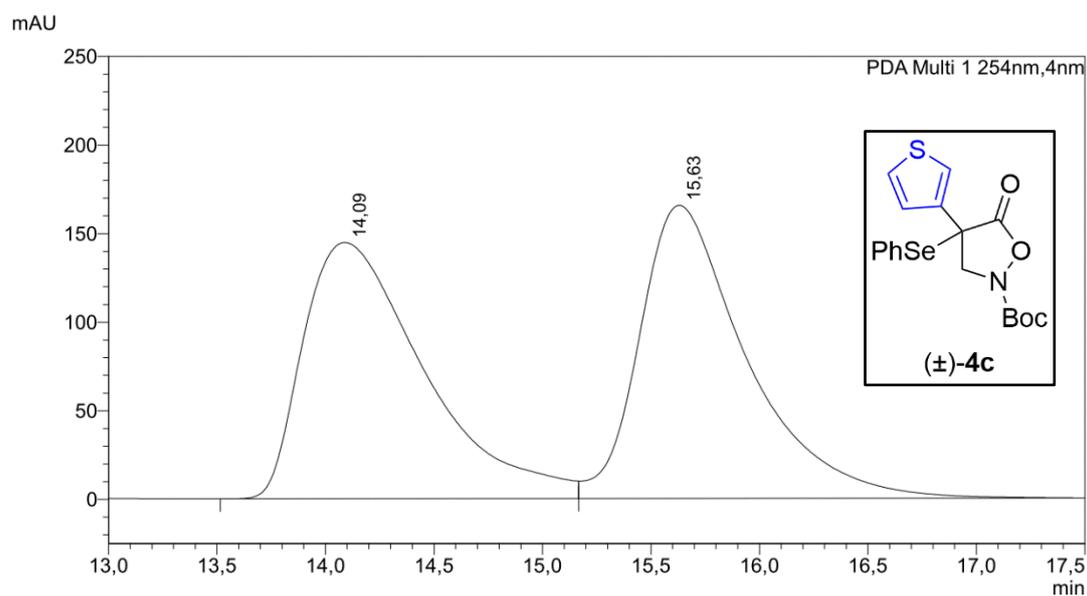
HPLC traces of **4c** (Chiralpak AD-H, *n*-hexane:*i*-PrOH = 20:1, flow rate 1.0 mL/min, 20 °C, $\lambda = 254$ nm)



Peak Table

PDA Ch1 254nm

Peak#	Ret. Time	Area	Area%
1	14,08	1708423	23,54
2	15,73	5549356	76,46
Total		7257779	100,00

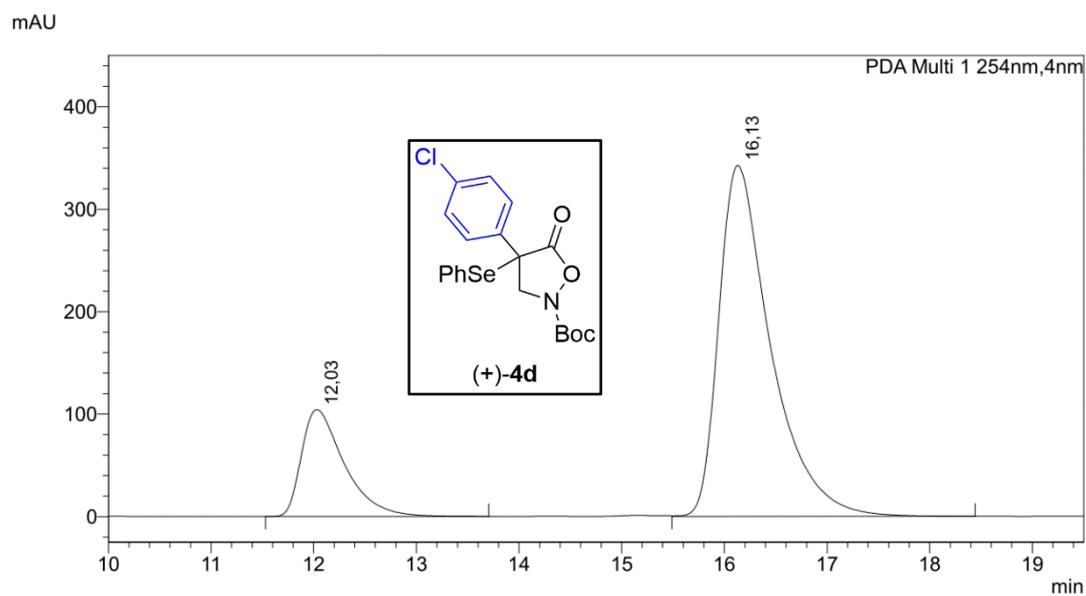


Peak Table

PDA Ch1 254nm

Peak#	Ret. Time	Area	Area%
1	14,09	5615054	49,71
2	15,63	5680643	50,29
Total		11295697	100,00

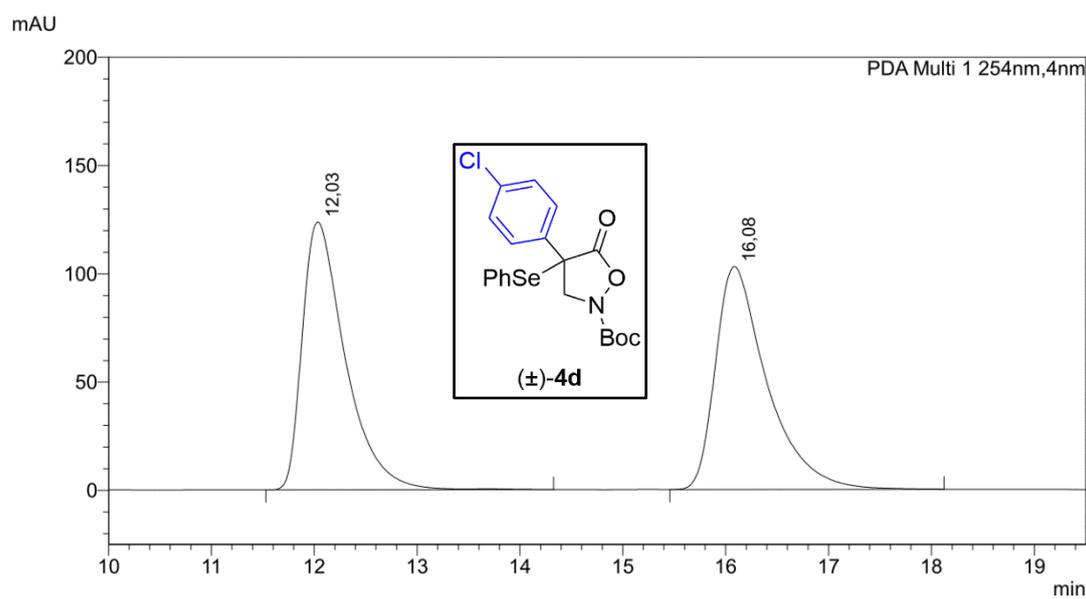
HPLC traces of **4d** (Chiralpak AD-H, *n*-hexane:*i*-PrOH = 20:1, flow rate 1.0 mL/min, 20 °C, $\lambda = 254$ nm)



Peak Table

PDA Ch1 254nm

Peak#	Ret. Time	Area	Area%
1	12,03	3020050	20,07
2	16,13	12029062	79,93
Total		15049112	100,00

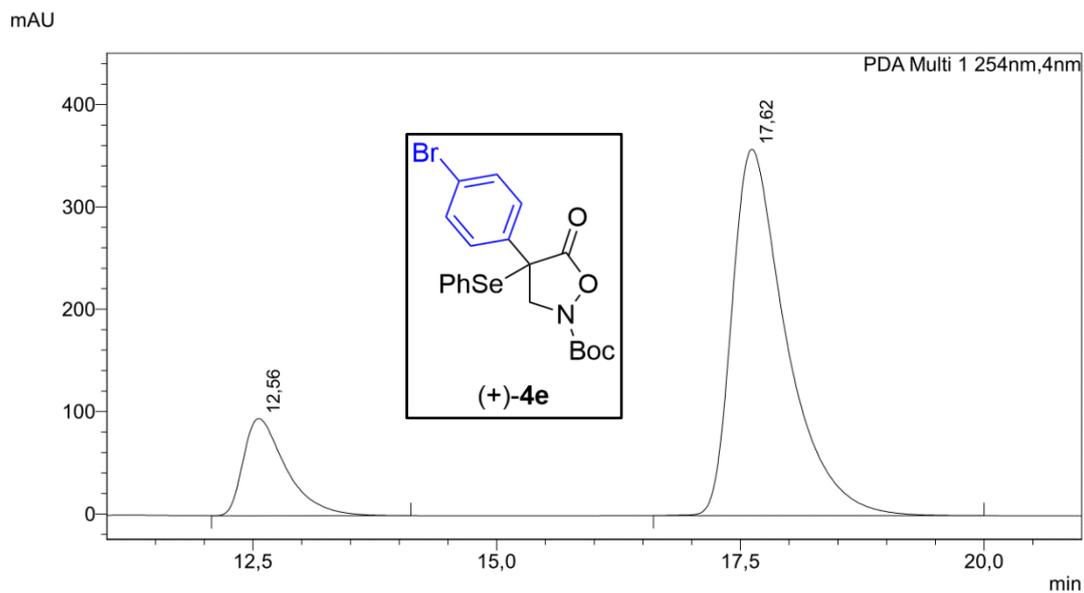


Peak Table

PDA Ch1 254nm

Peak#	Ret. Time	Area	Area%
1	12,03	3589177	50,06
2	16,08	3580893	49,94
Total		7170070	100,00

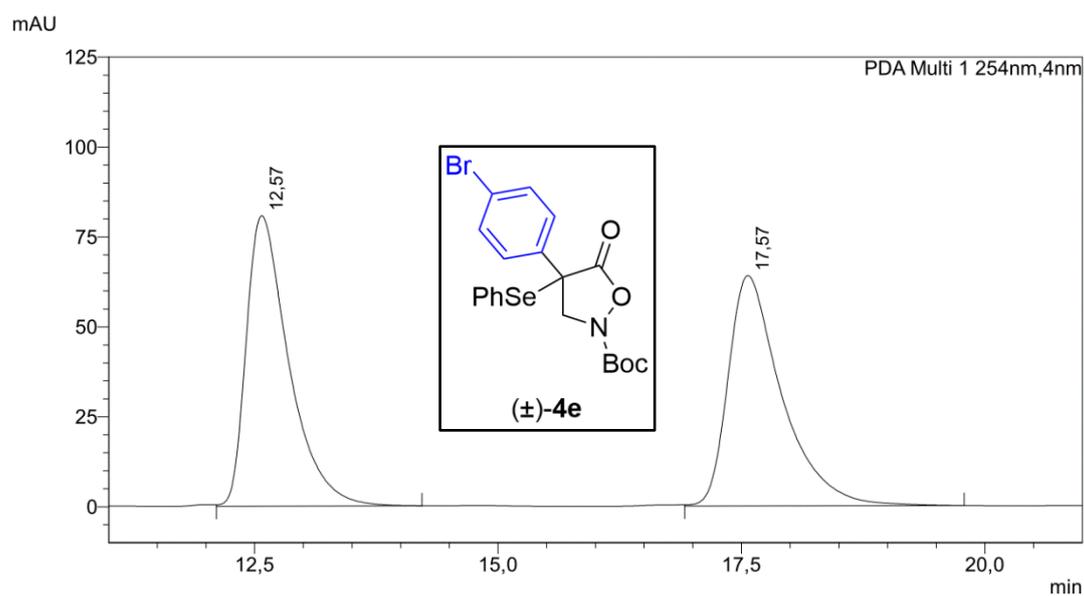
HPLC traces of **4e** (Chiralpak AD-H, *n*-hexane:*i*-PrOH = 20:1, flow rate 1.0 mL/min, 20 °C, $\lambda = 254$ nm)



Peak Table

PDA Ch1 254nm

Peak#	Ret. Time	Area	Area%
1	12,56	2901606	17,29
2	17,62	13884221	82,71
Total		16785827	100,00

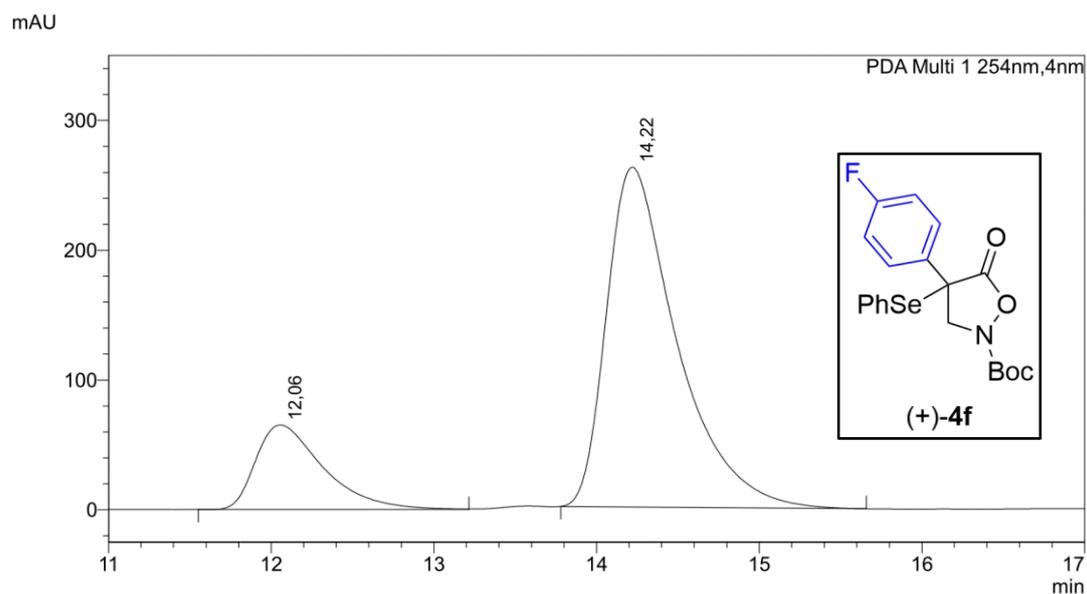


Peak Table

PDA Ch1 254nm

Peak#	Ret. Time	Area	Area%
1	12,57	2453924	49,97
2	17,57	2457074	50,03
Total		4910998	100,00

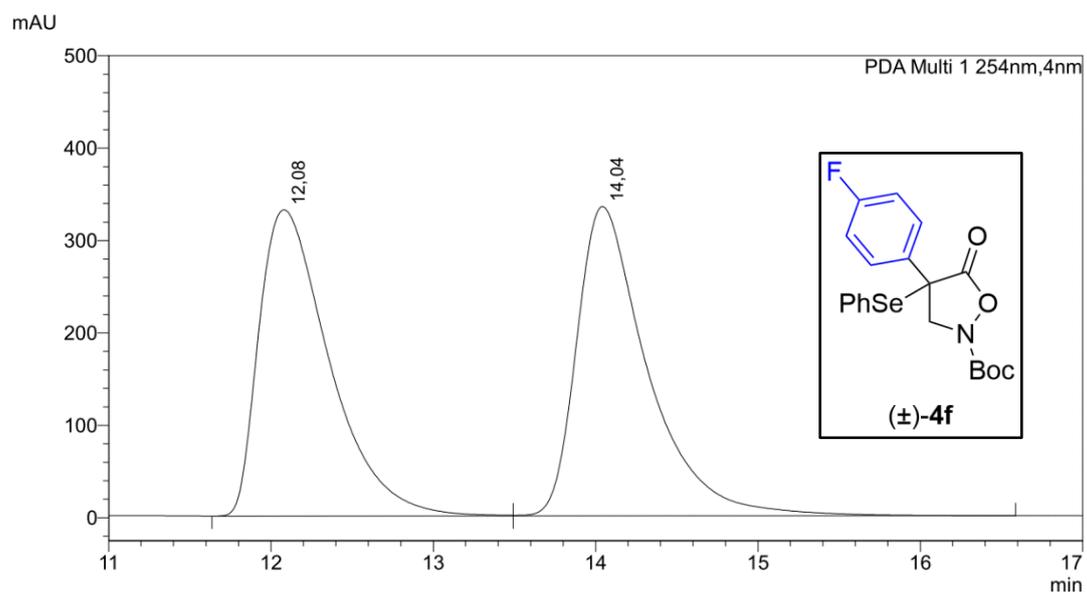
HPLC traces of **4f** (Chiralpak AD-H, *n*-hexane:*i*-PrOH = 20:1, flow rate 1.0 mL/min, 20 °C, $\lambda = 254$ nm)



Peak Table

PDA Ch1 254nm

Peak#	Ret. Time	Area	Area%
1	12,06	1879654	19,26
2	14,22	7878156	80,74
Total		9757810	100,00

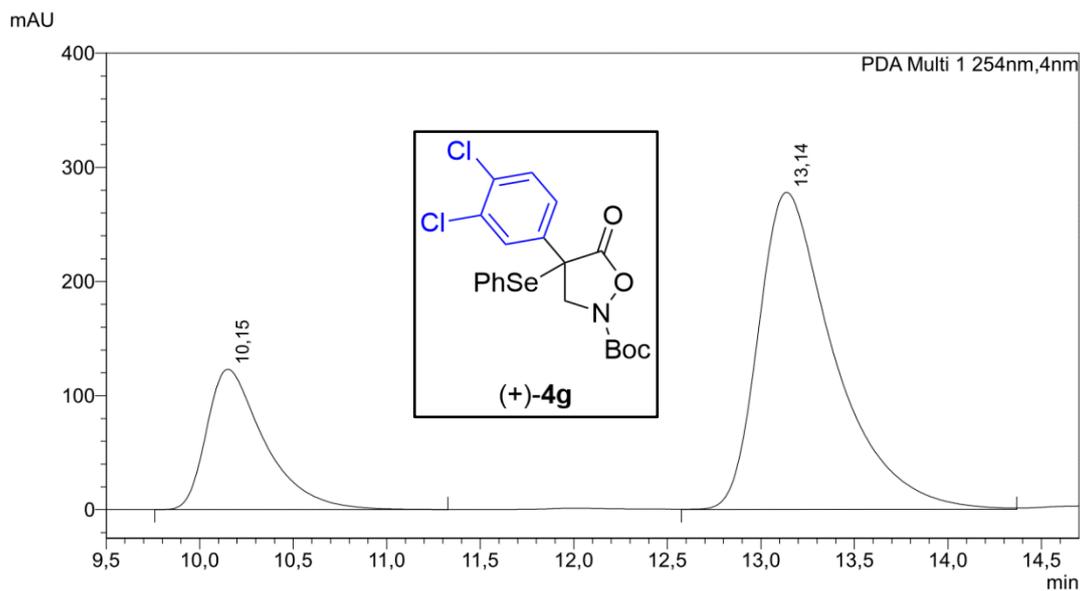


Peak Table

PDA Ch1 254nm

Peak#	Ret. Time	Area	Area%
1	12,08	10007146	49,53
2	14,04	10198474	50,47
Total		20205620	100,00

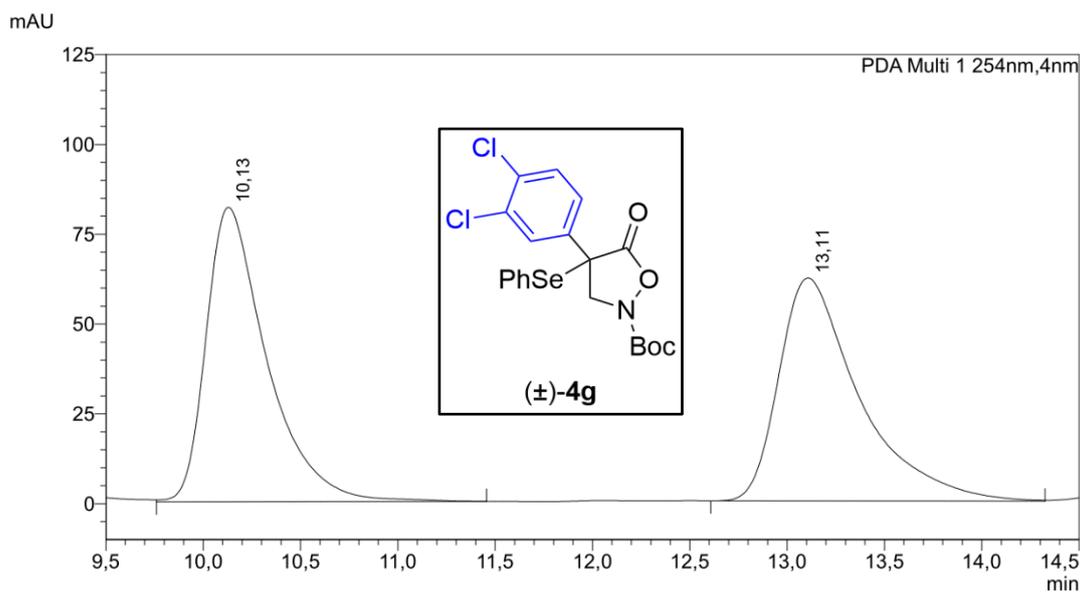
HPLC traces of **4g** (Chiralpak AD-H, *n*-hexane:*i*-PrOH = 20:1, flow rate 1.0 mL/min, 20 °C, $\lambda = 254$ nm)



Peak Table

PDA Ch1 254nm

Peak#	Ret. Time	Area	Area%
1	10,15	2654198	25,22
2	13,14	7869025	74,78
Total		10523223	100,00

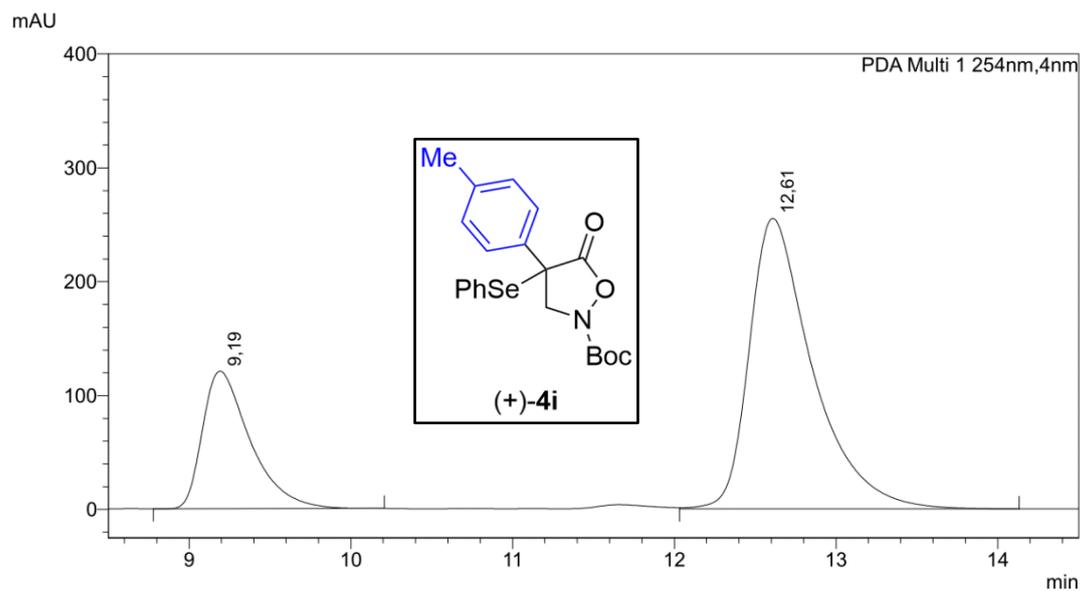


Peak Table

PDA Ch1 254nm

Peak#	Ret. Time	Area	Area%
1	10,13	1775586	49,89
2	13,11	1783726	50,11
Total		3559312	100,00

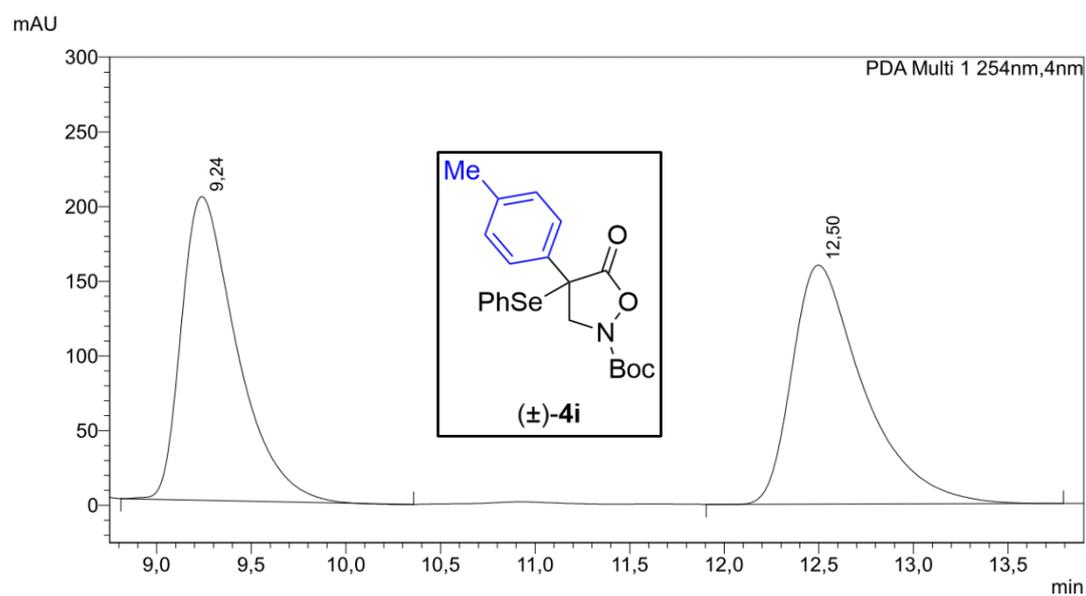
HPLC traces of **4i** (Chiralpak AD-H, *n*-hexane:*i*-PrOH = 20:1, flow rate 1.0 mL/min, 20 °C, $\lambda = 254$ nm)



Peak Table

PDA Ch1 254nm

Peak#	Ret. Time	Area	Area%
1	9,19	2471161	26,27
2	12,61	6934762	73,73
Total		9405924	100,00

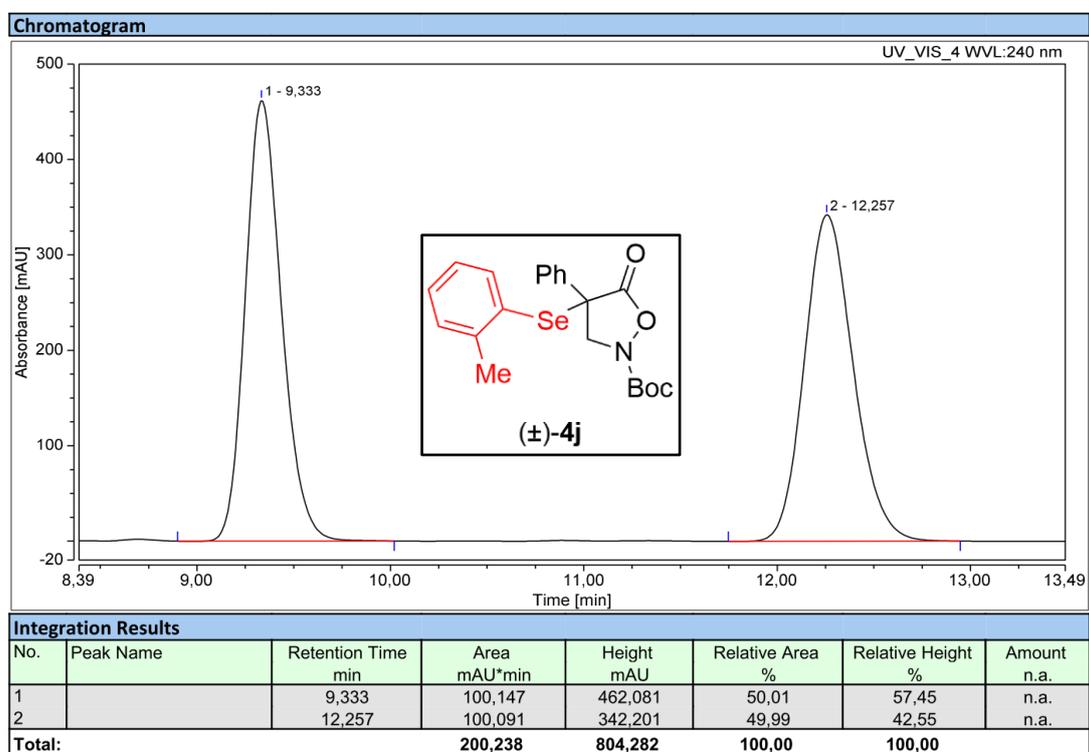
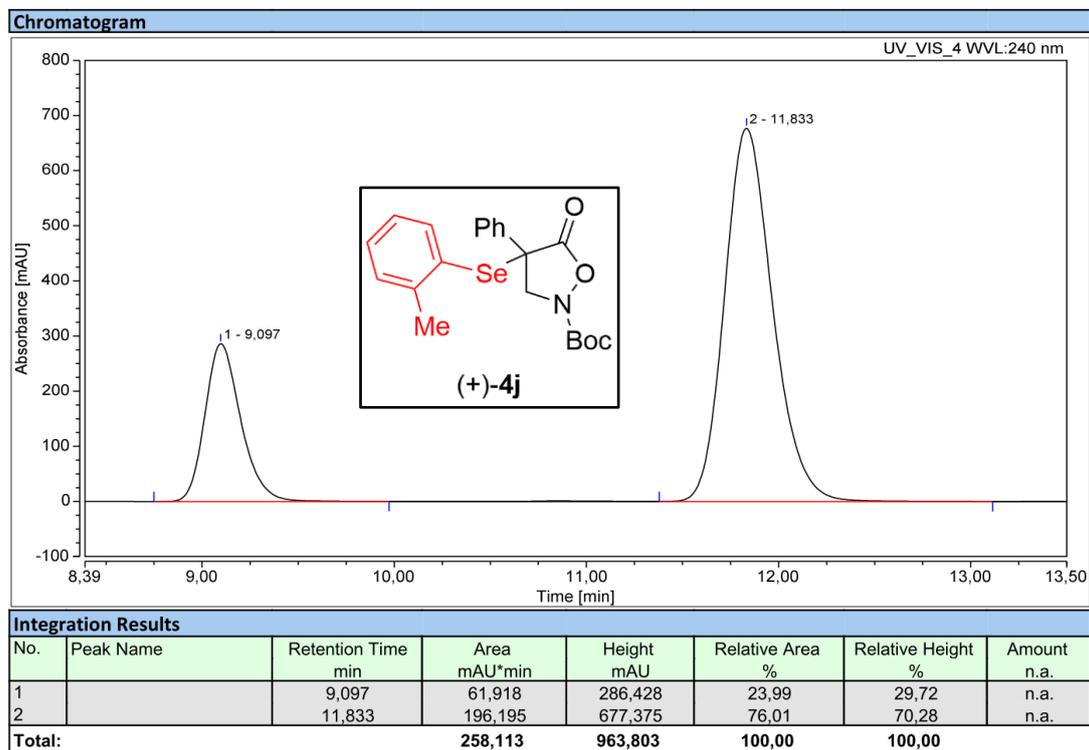


Peak Table

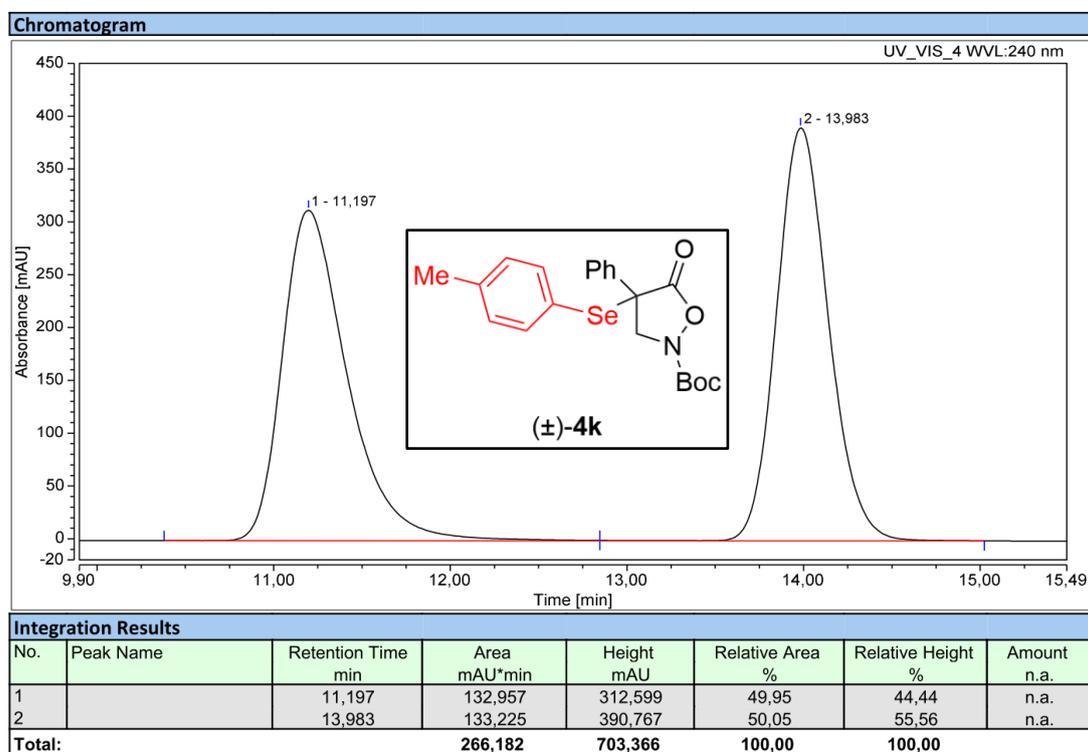
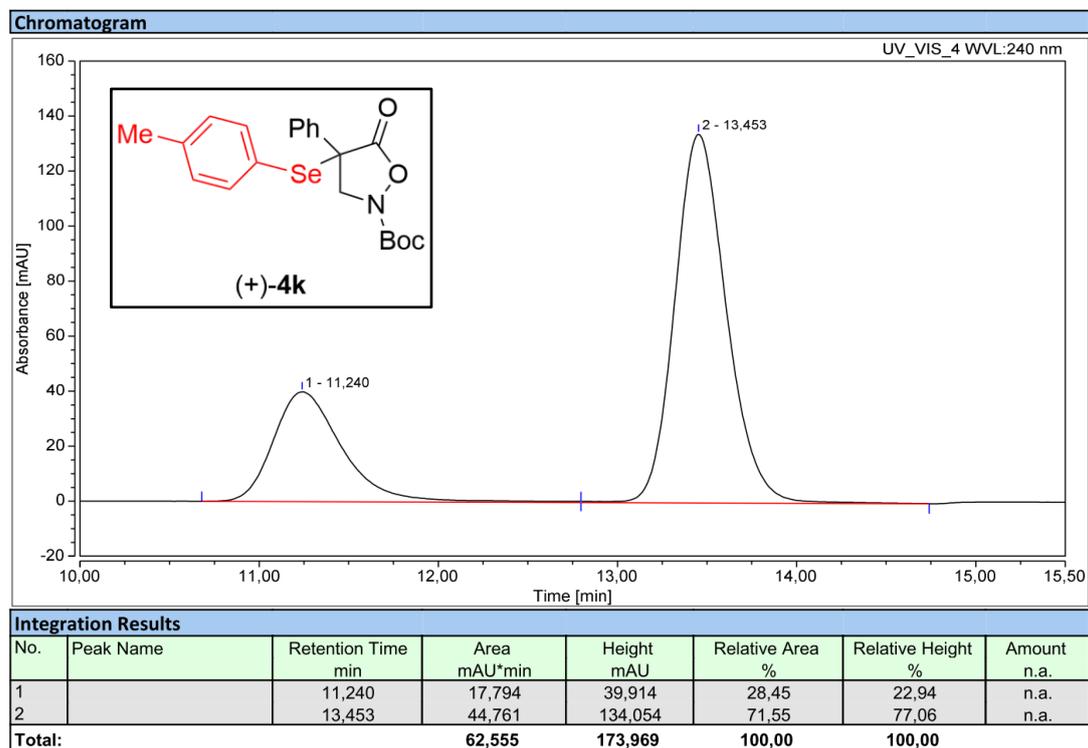
PDA Ch1 254nm

Peak#	Ret. Time	Area	Area%
1	9,24	4142184	49,61
2	12,50	4207357	50,39
Total		8349541	100,00

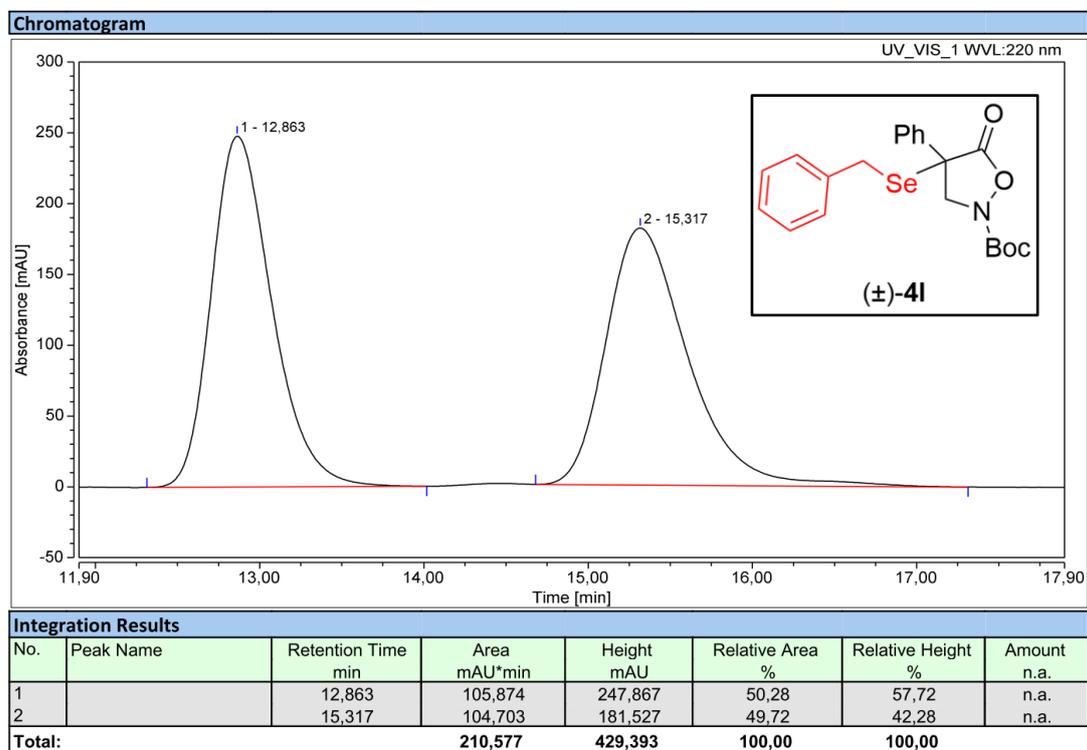
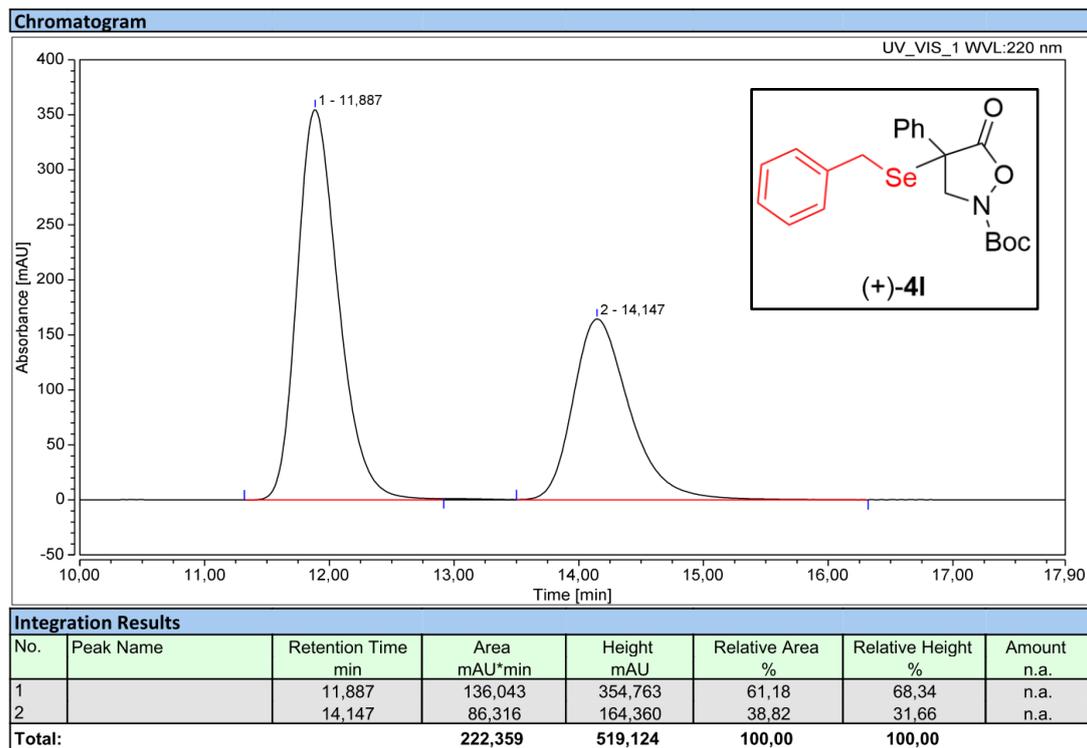
HPLC traces of **4j** (Chiralpak AD-H, *n*-hexane:*i*-PrOH = 20:1, flow rate 1.0 mL/min, 10 °C, $\lambda = 240$ nm)



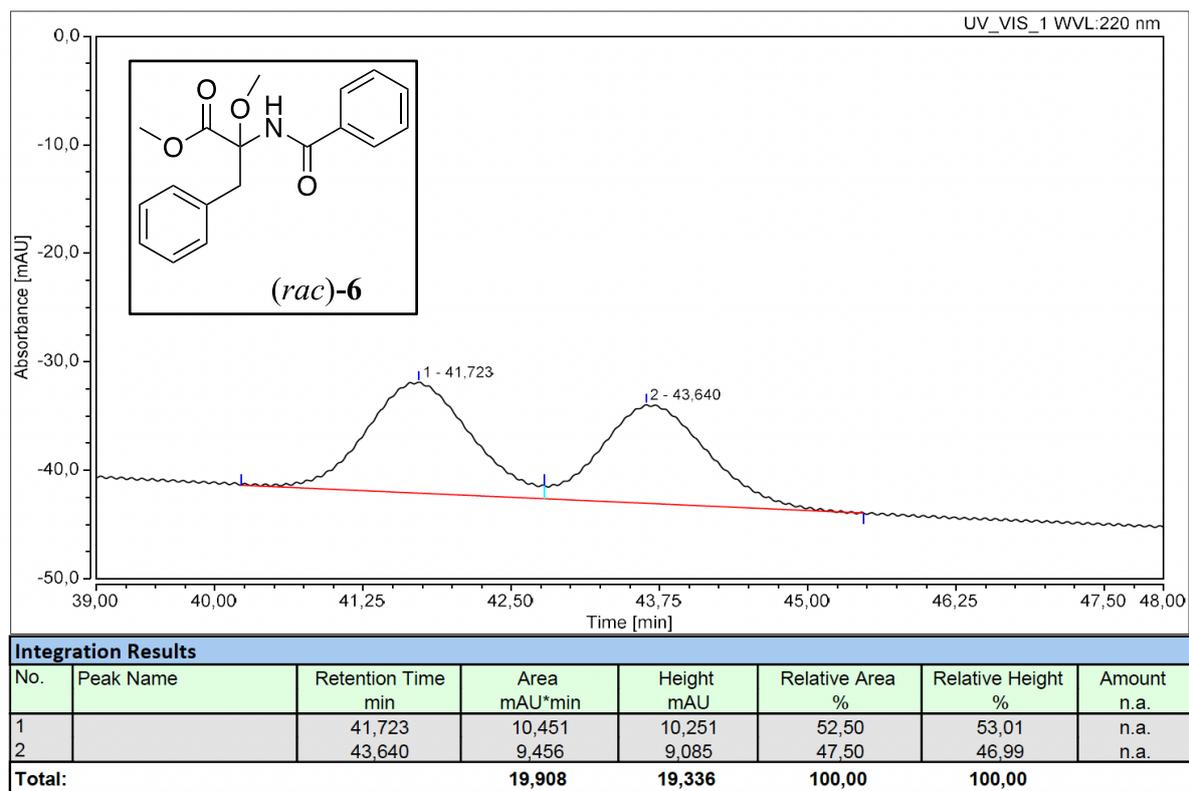
HPLC traces of **4k** (Chiralpak AD-H, *n*-hexane:*i*-PrOH = 20:1, flow rate 1.0 mL/min, 10 °C, $\lambda = 240$ nm)



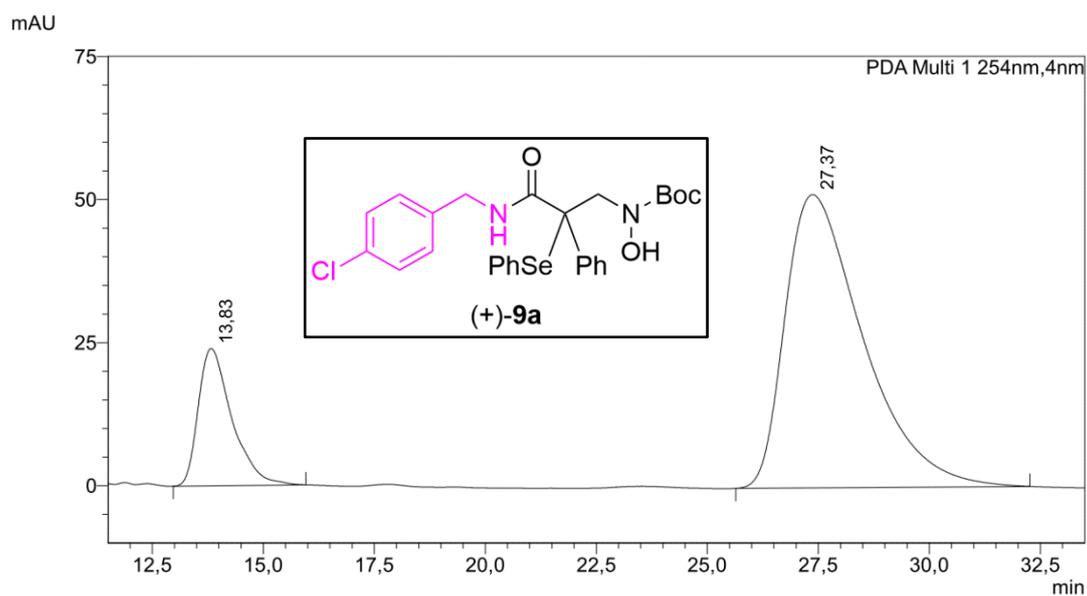
HPLC traces of **4I** (Chiralpak OD-H, *n*-hexane:*i*-PrOH = 20:1, flow rate 1.0 mL/min, 10 °C, $\lambda = 220$ nm)



HPLC traces of **6** (Chiralpak AD-H, *n*-hexane:*i*-PrOH = 20:1, flow rate 0.9 mL/min, 10 °C, $\lambda = 220$ nm)



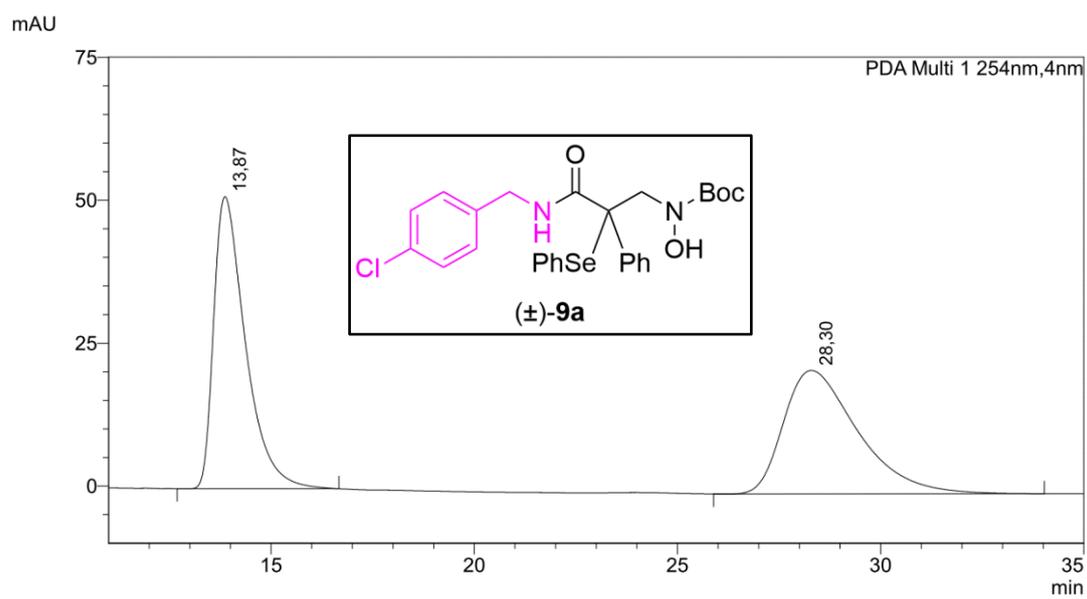
HPLC traces of **9a** (Chiralpak OD-H, *n*-hexane:*i*-PrOH = 2:1, flow rate 0.5 mL/min, 20 °C, $\lambda = 254$ nm)



Peak Table

PDA Ch1 254nm

Peak#	Ret. Time	Area	Area%
1	13,83	1264252	16,81
2	27,37	6256611	83,19
Total		7520863	100,00

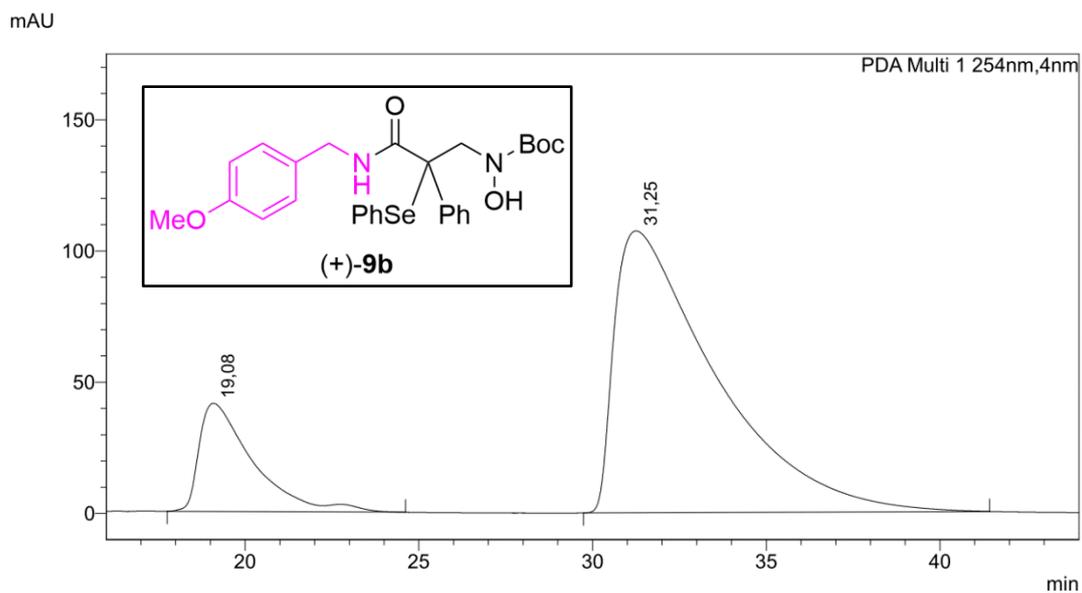


Peak Table

PDA Ch1 254nm

Peak#	Ret. Time	Area	Area%
1	13,87	2774971	50,27
2	28,30	2744791	49,73
Total		5519762	100,00

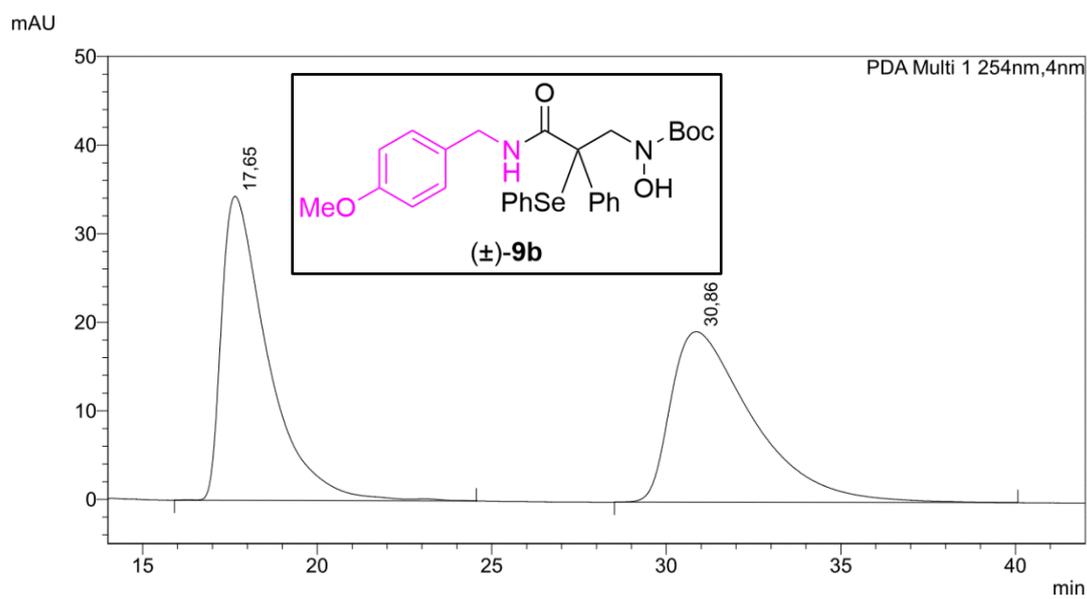
HPLC traces of **9b** (Chiralpak OD-H, *n*-hexane:*i*-PrOH = 2:1, flow rate 0.5 mL/min, 20 °C, $\lambda = 254$ nm)



Peak Table

PDA Ch1 254nm

Peak#	Ret. Time	Area	Area%
1	19,08	4458318	17,04
2	31,25	21703836	82,96
Total		26162154	100,00

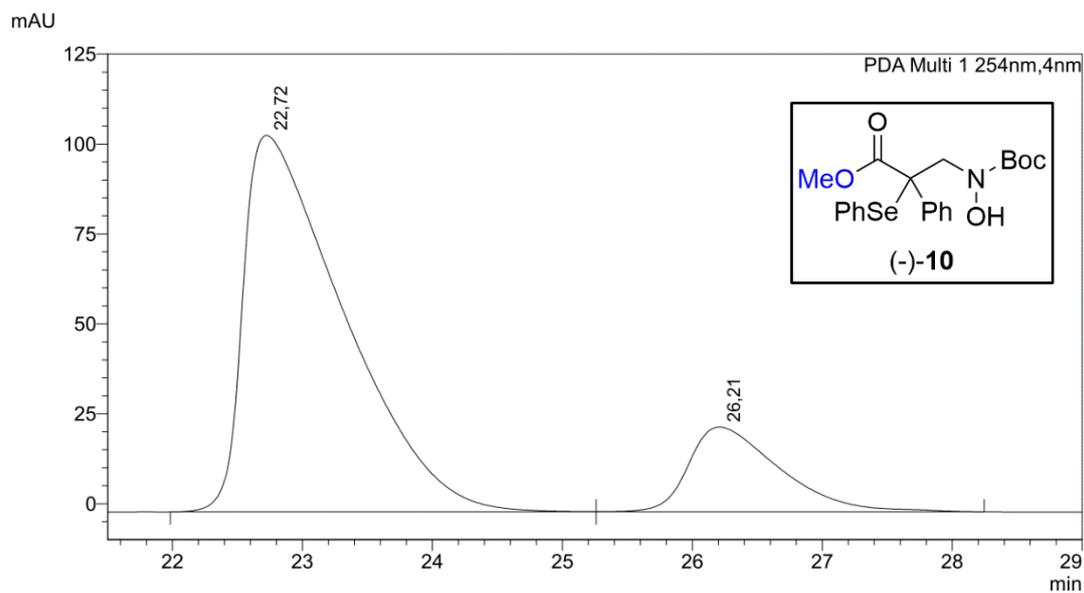


Peak Table

PDA Ch1 254nm

Peak#	Ret. Time	Area	Area%
1	17,65	3164909	50,19
2	30,86	3140800	49,81
Total		6305709	100,00

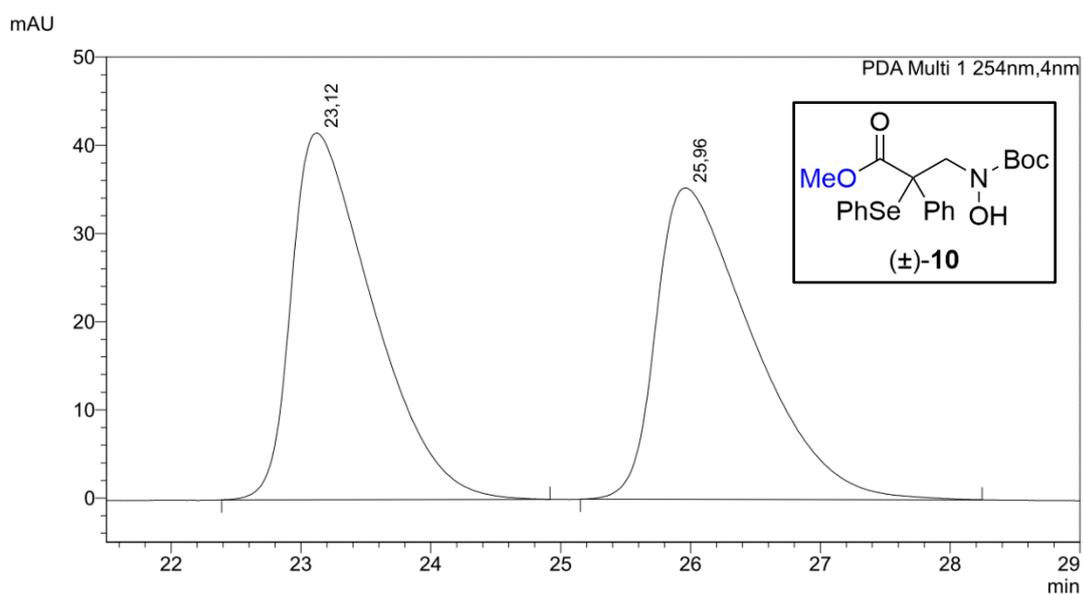
HPLC traces of **10** (YMC Chiral ART Cellulose-SB, *n*-hexane:*i*-PrOH = 4:1, flow rate 0.5 mL/min, 20 °C, λ = 254 nm)



Peak Table

PDA Ch1 254nm

Peak#	Ret. Time	Area	Area%
1	22,72	5628121	83,07
2	26,21	1147292	16,93
Total		6775414	100,00



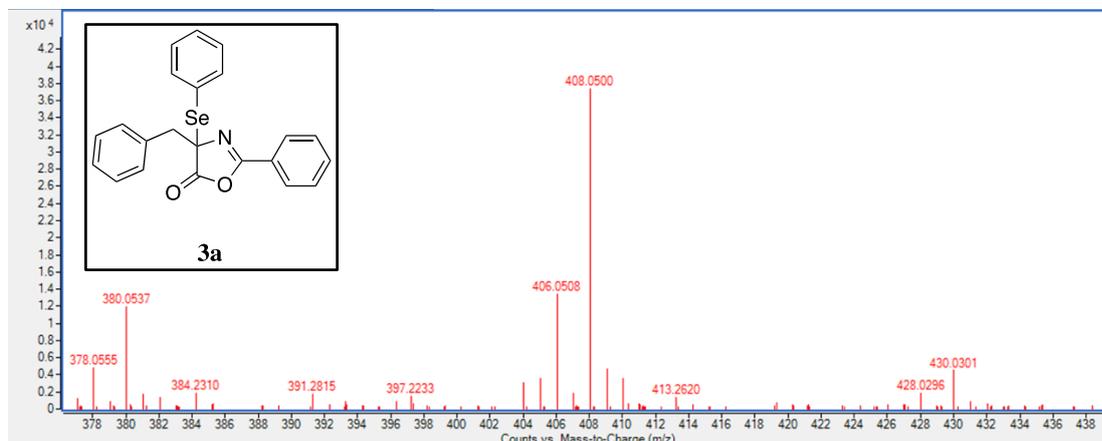
Peak Table

PDA Ch1 254nm

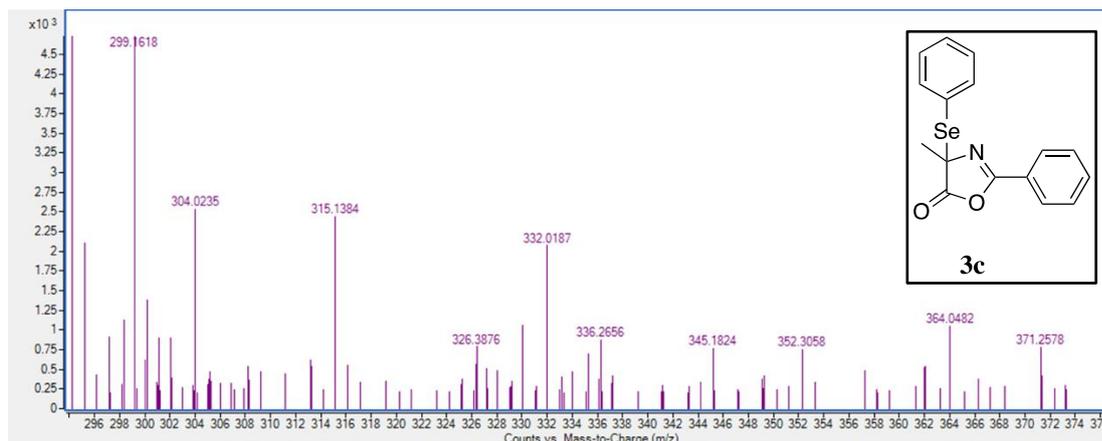
Peak#	Ret. Time	Area	Area%
1	23,12	1816104	49,96
2	25,96	1818670	50,04
Total		3634774	100,00

11. HRMS Data

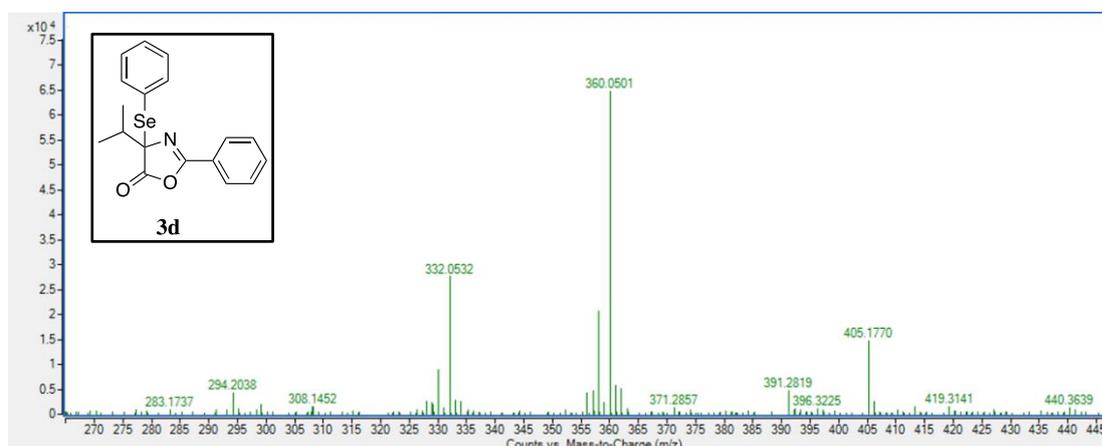
HRMS spectrum of 3a (ESI-QTOF, MeOH) m/z : $[M+H]^+$ calculated for $C_{23}H_{17}NO_2Se$, 408.0498; found, 408.0500.



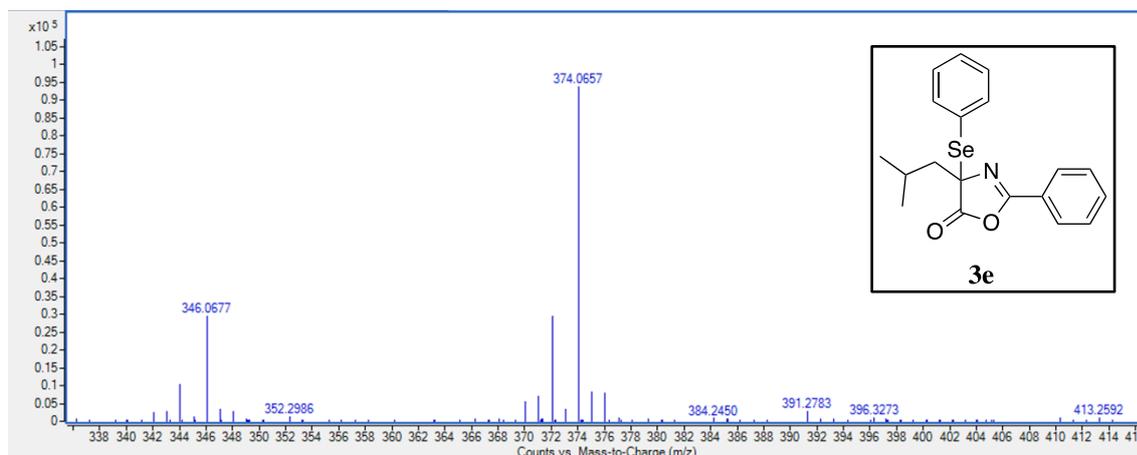
HRMS spectrum of 3c (ESI-QTOF, MeOH) m/z : $[M+H]^+$ calculated for $C_{16}H_{14}NO_2Se$, 332.0185; found, 332.0187.



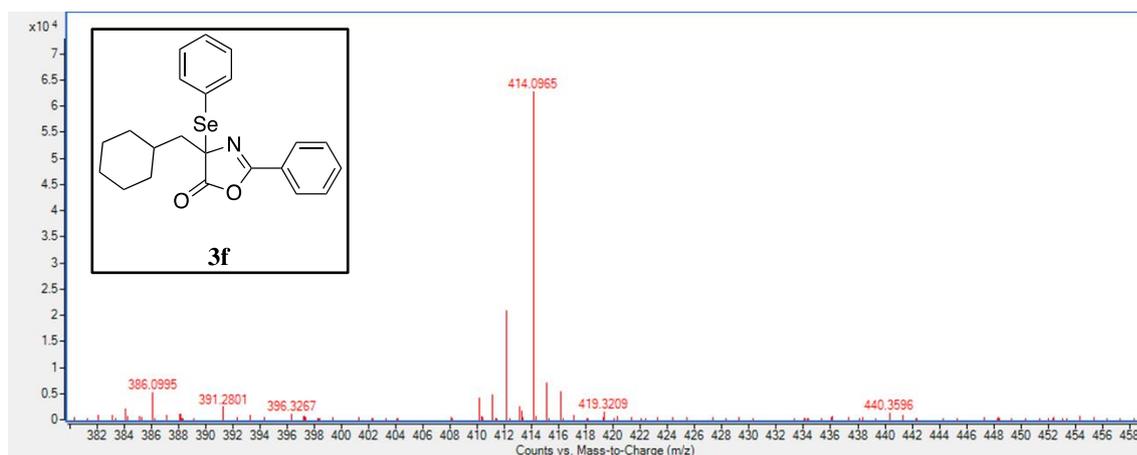
HRMS spectrum of 3d (ESI-QTOF, MeOH) m/z : $[M+H]^+$ calculated for $C_{18}H_{18}NO_2Se$, 360.0498; found, 360.0501.



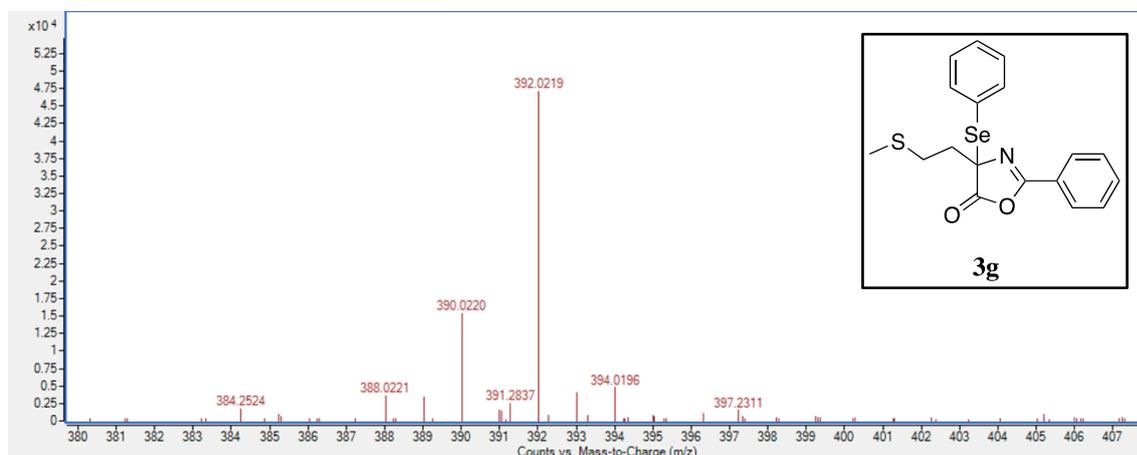
HRMS spectrum of 3e (ESI-QTOF, MeOH) m/z : $[M+H]^+$ calculated for $C_{19}H_{20}NO_2Se$, 374.0654; found, 374.0657.



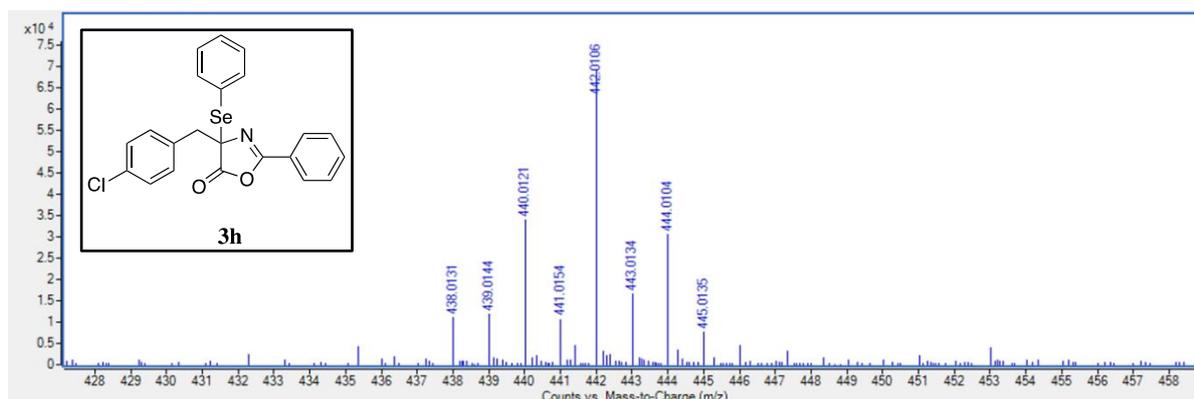
HRMS spectrum of 3f (ESI-QTOF, MeOH) m/z : $[M+H]^+$ calculated for $C_{22}H_{24}NO_2Se$, 414.0967; found, 414.0965.



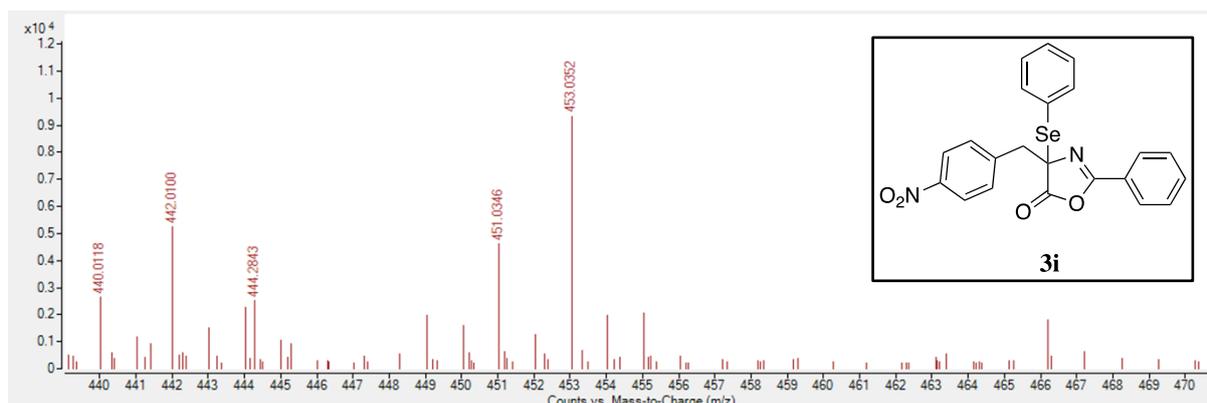
HRMS spectrum of 3g (ESI-QTOF, MeOH) m/z : $[M+H]^+$ calculated for $C_{18}H_{18}NO_2SSe$, 392.0218; found, 392.0219.



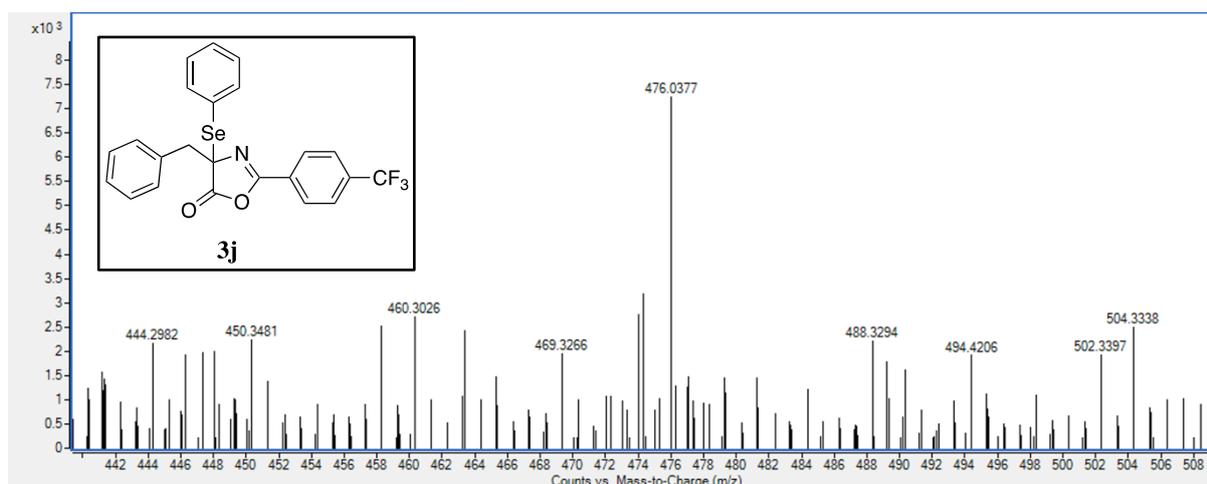
HRMS spectrum of 3h (ESI-QTOF, MeOH) m/z : $[M+H]^+$ calculated for $C_{22}H_{17}ClNO_2Se$, 442.0108; found, 442.0106.



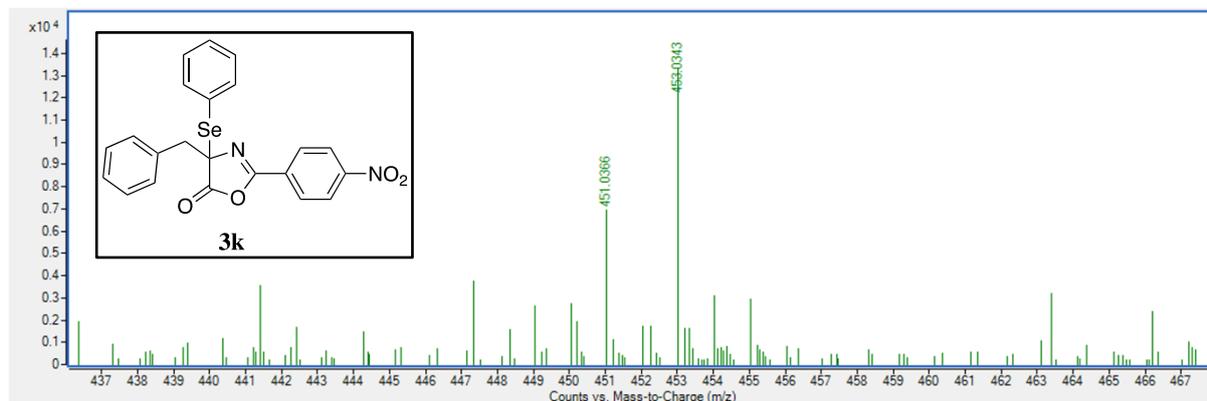
HRMS spectrum of 3i (ESI-QTOF, MeOH) m/z : $[M+H]^+$ calculated for $C_{22}H_{17}N_2O_4Se$, 453.0348; found, 453.0352.



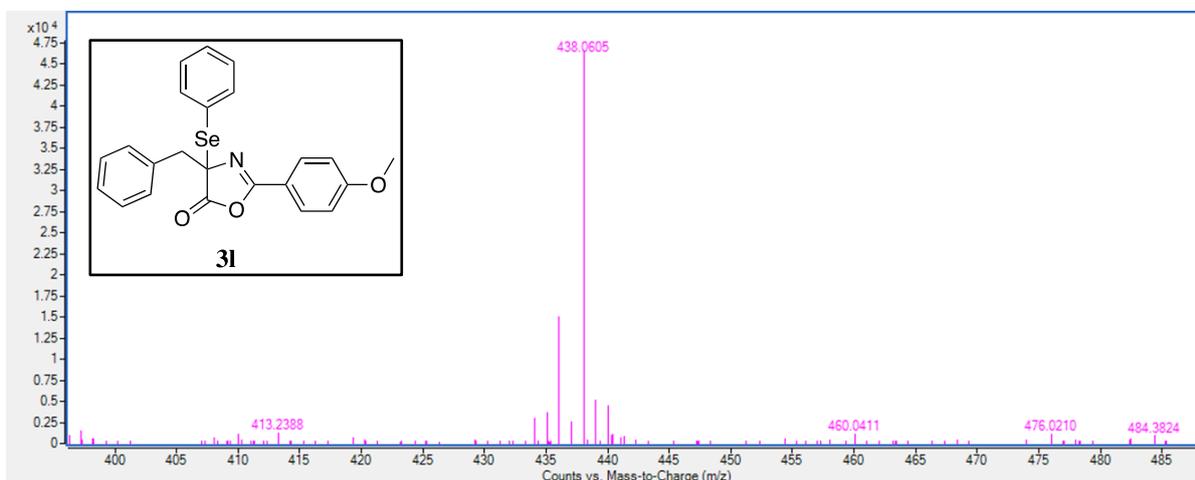
HRMS spectrum of 3j (ESI-QTOF, MeOH) m/z : $[M+H]^+$ calculated for $C_{23}H_{17}F_3NO_2Se$, 476.0371; found, 476.0377.



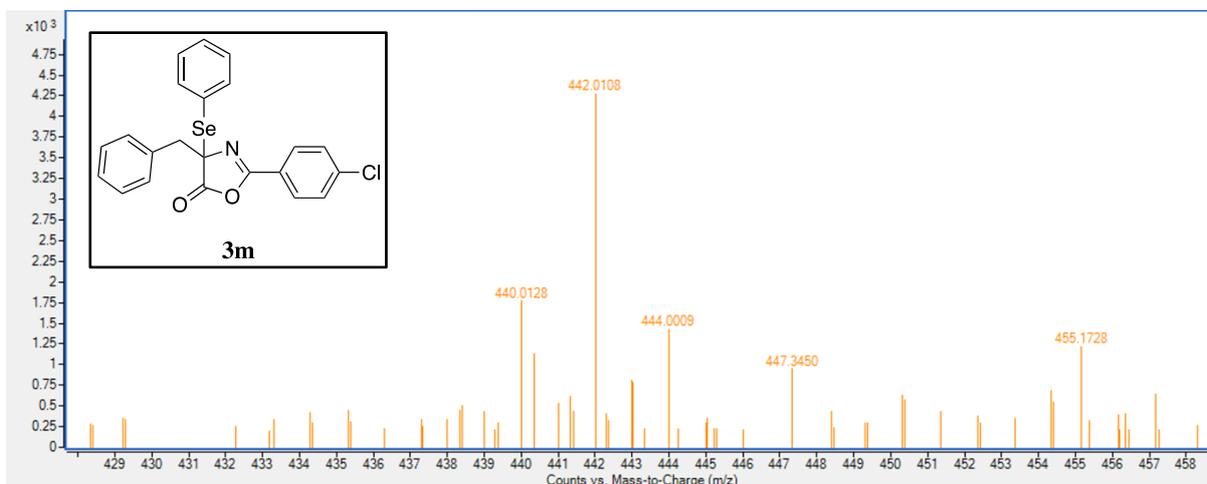
HRMS spectrum of 3k (ESI-QTOF, MeOH) m/z : $[M+H]^+$ calculated for $C_{22}H_{17}N_2O_4Se$, 453.0348; found, 453.0343.



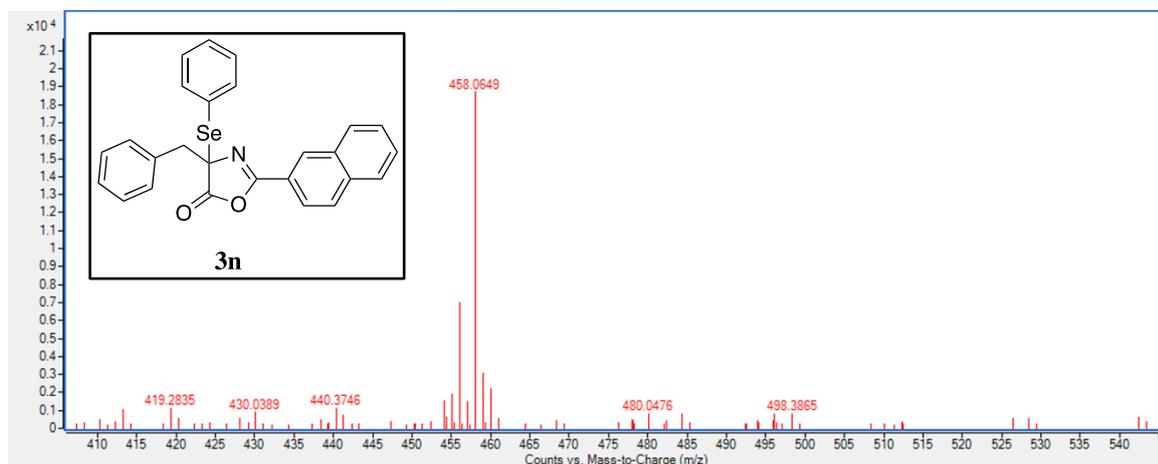
HRMS spectrum of 3l (ESI-QTOF, MeOH) m/z : $[M+H]^+$ calculated for $C_{23}H_{20}NO_3Se$, 438.0603; found, 438.0605.



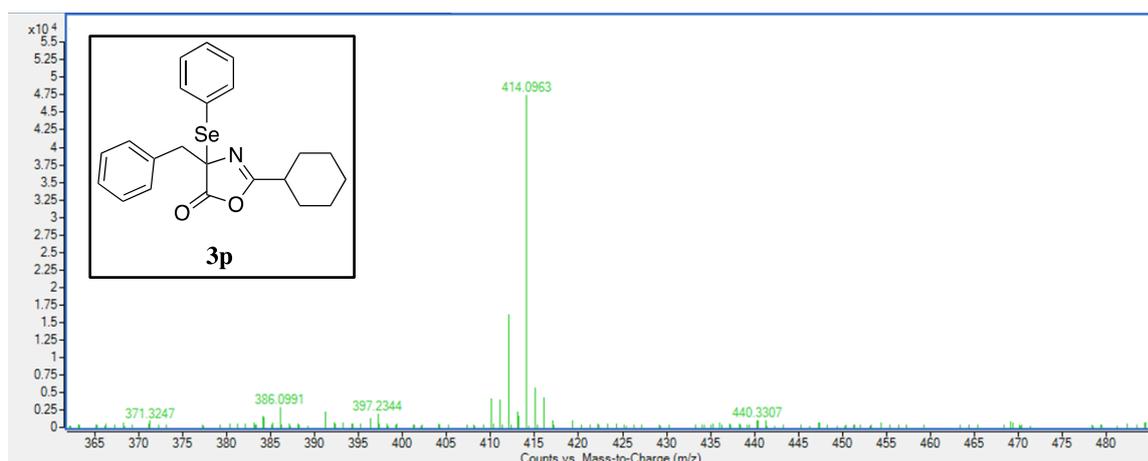
HRMS spectrum of 3m (ESI-QTOF, MeOH) m/z : $[M+H]^+$ calculated for $C_{22}H_{17}ClNO_2Se$, 442.0108; found, 442.0108.



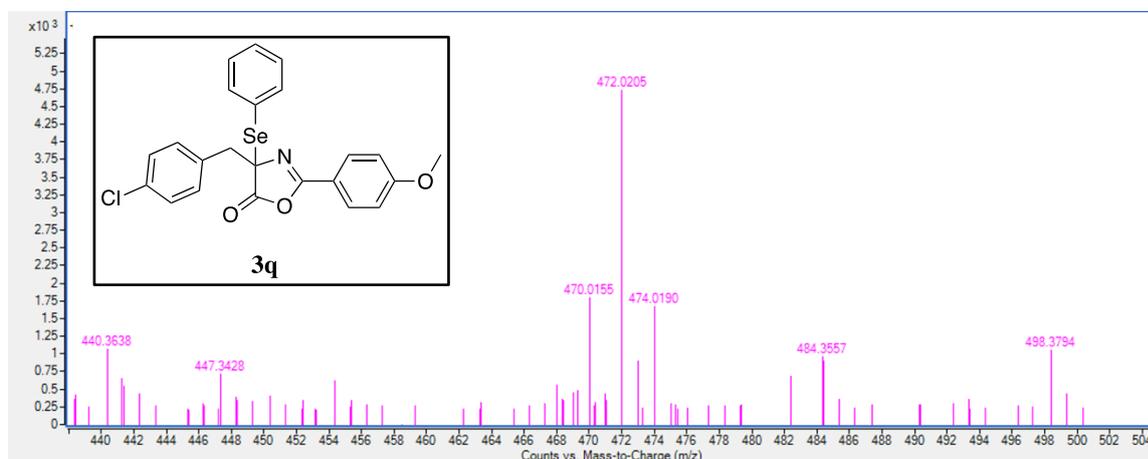
HRMS spectrum of 3n (ESI-QTOF, MeOH) m/z : $[M+H]^+$ calculated for $C_{26}H_{20}NO_2Se$, 458.0654; found, 458.0649.



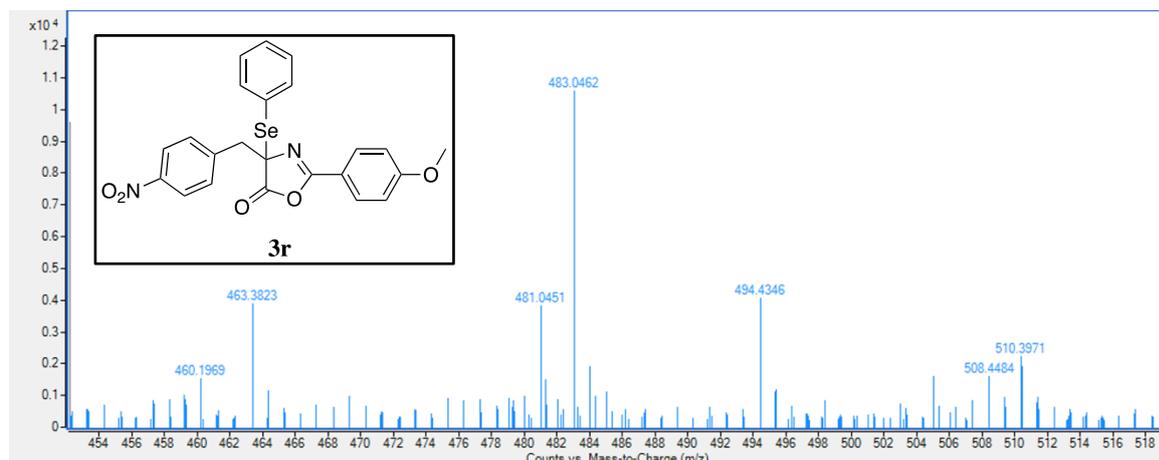
HRMS spectrum of 3p (ESI-QTOF, MeOH) m/z : $[M+H]^+$ calculated for $C_{22}H_{24}NO_2Se$, 414.0967; found, 414.0963.



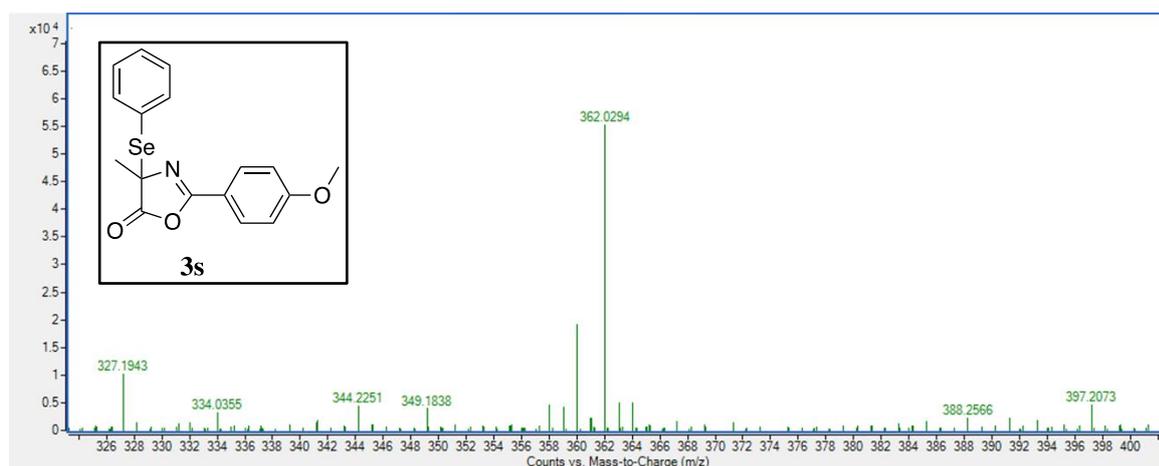
HRMS spectrum of 3q (ESI-QTOF, MeOH) m/z : $[M+H]^+$ calculated for $C_{23}H_{19}ClNO_3Se$, 472.0213; found, 472.0205.



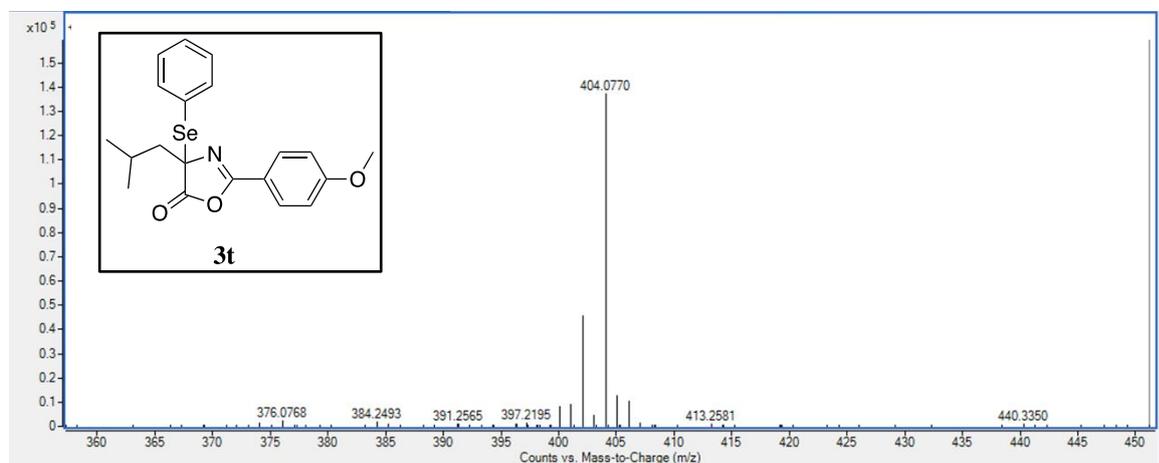
HRMS spectrum of 3r (ESI-QTOF, MeOH) m/z : $[M+H]^+$ calculated for $C_{23}H_{19}N_2O_5Se$, 483.0454; found, 483.0462.



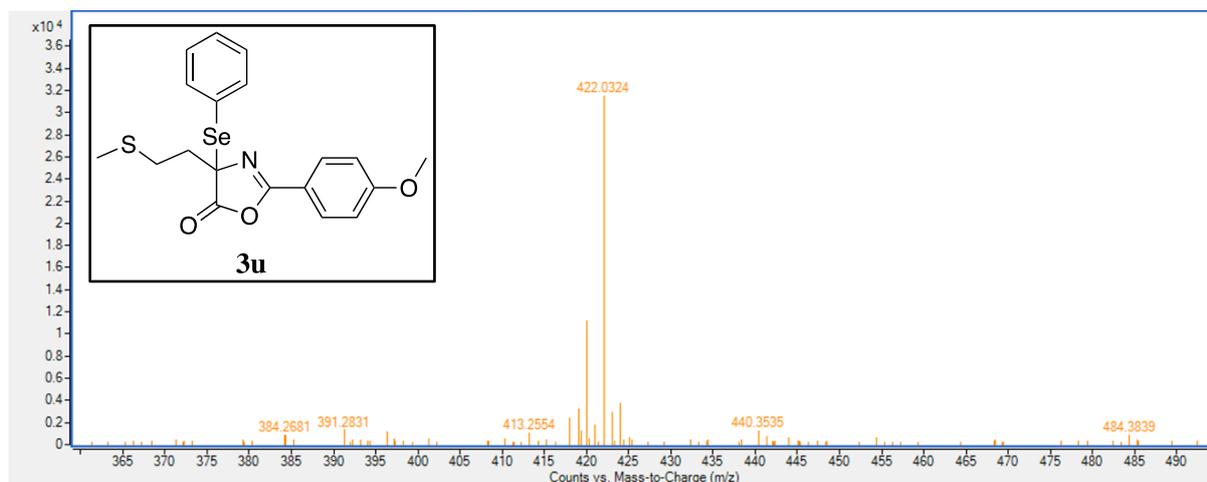
HRMS spectrum of 3s (ESI-QTOF, MeOH) m/z : $[M+H]^+$ calculated for $C_{17}H_{16}NO_3Se$, 362.0290; found, 362.0294.



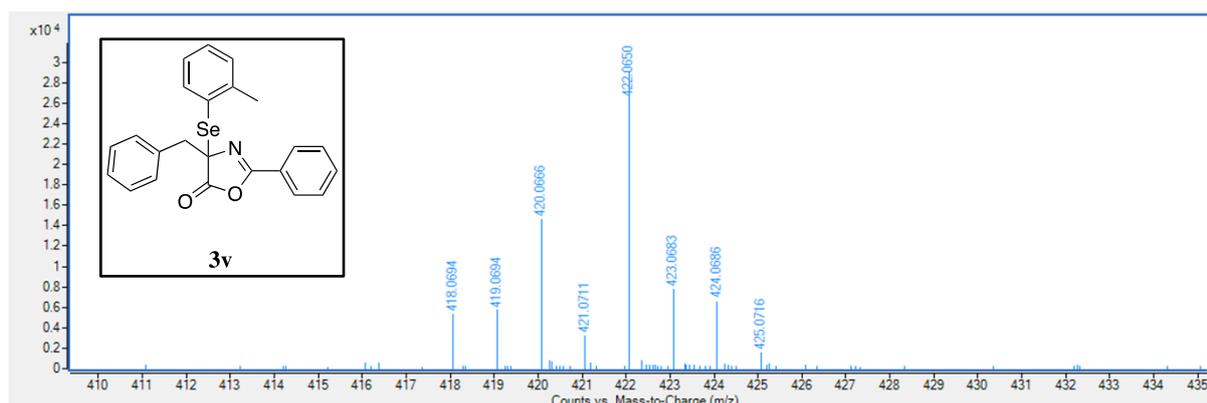
HRMS spectrum of 3t (ESI-QTOF, MeOH) m/z : $[M+H]^+$ calculated for $C_{20}H_{22}NO_3Se$, 404.0760; found, 404.0770.



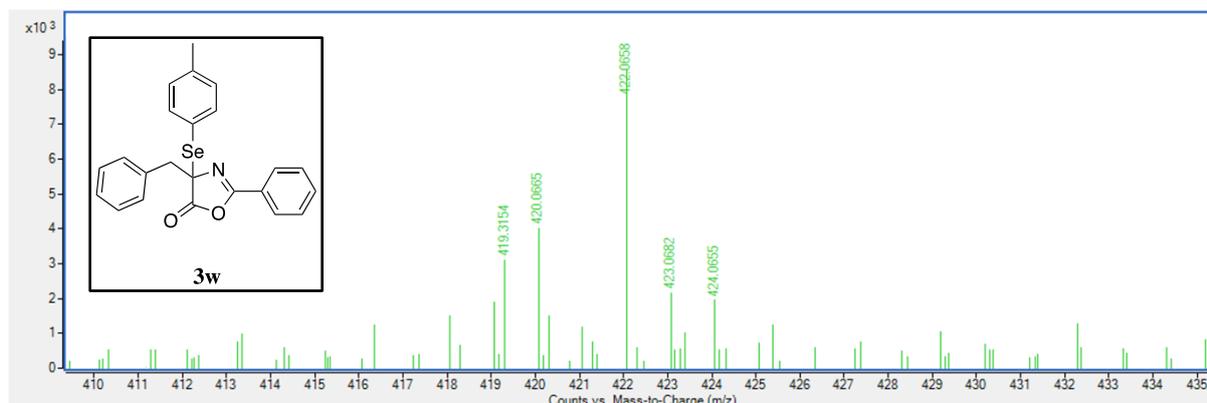
HRMS spectrum of 3u (ESI-QTOF, MeOH) m/z : $[M+H]^+$ calculated for $C_{19}H_{20}NO_3SSe$, 422.0324; found, 422.0324.



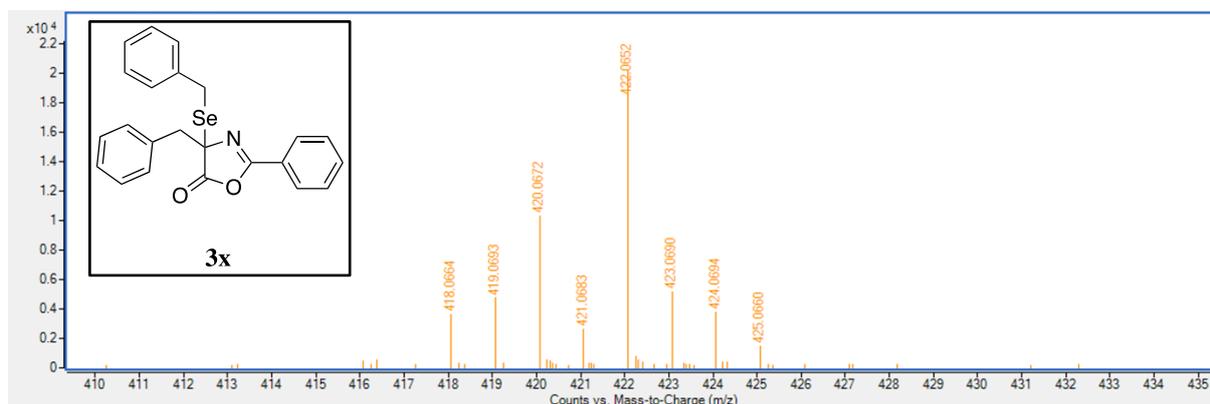
HRMS spectrum of 3v (ESI-QTOF, MeOH) m/z : $[M+H]^+$ calculated for $C_{23}H_{20}NO_2Se$, 422.0654; found, 422.0650.



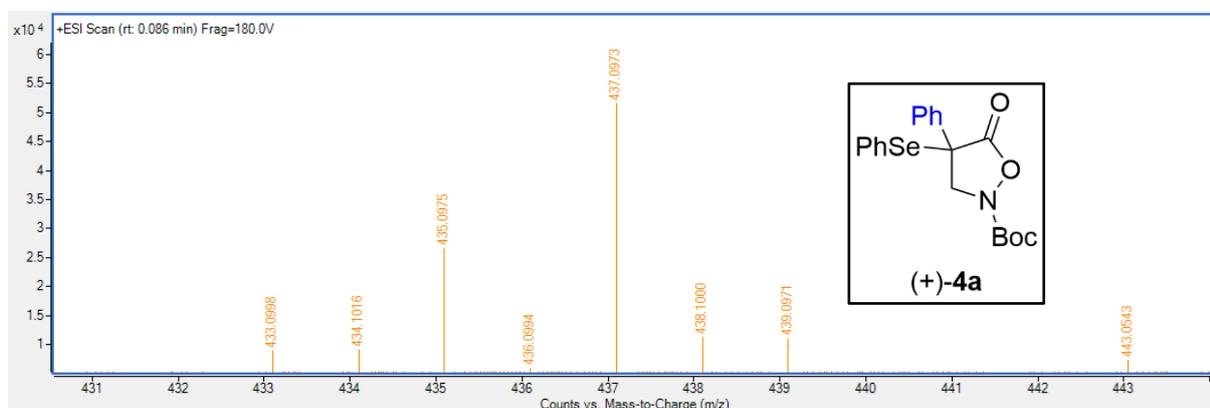
HRMS spectrum of 3w (ESI-QTOF, MeOH) m/z : $[M+H]^+$ calculated for $C_{23}H_{20}NO_2Se$, 422.0654; found, 422.0658.



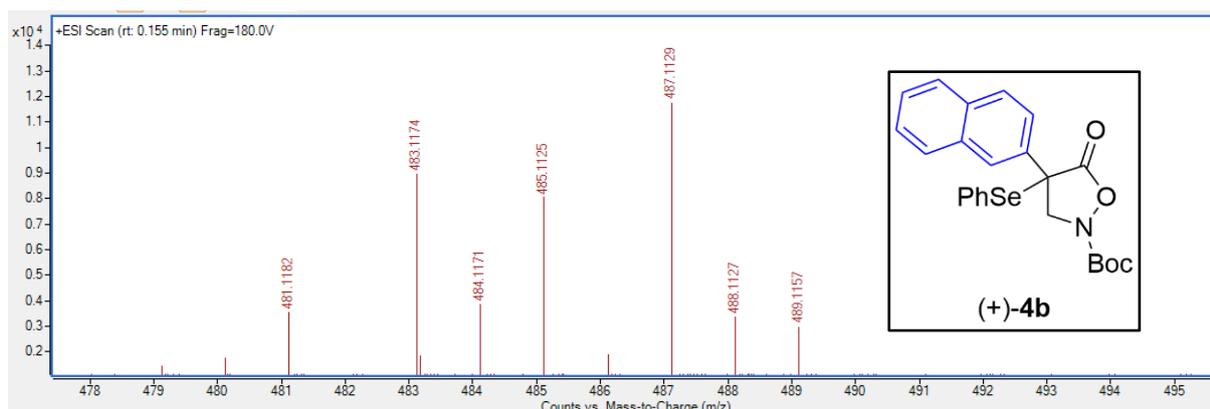
HRMS spectrum of 3x (ESI-QTOF, MeOH) m/z : $[M+H]^+$ calculated for $C_{23}H_{20}NO_2Se$, 422.0654; found, 422.0652.



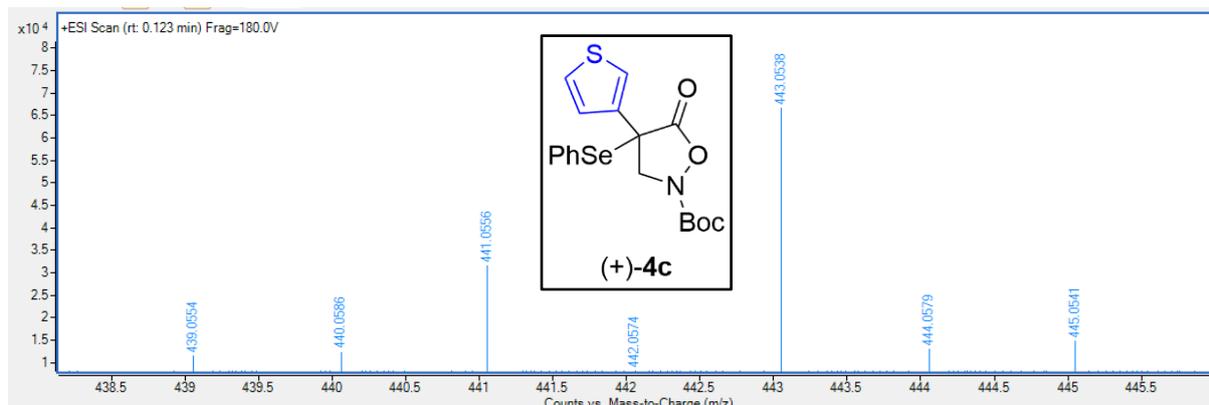
HRMS spectrum of 4a (ESI-QTOF, MeOH) m/z : $[M+NH_4]^+$ calculated for $C_{20}H_{25}N_2O_4Se^+$, 437.0974; found, 437.0973.



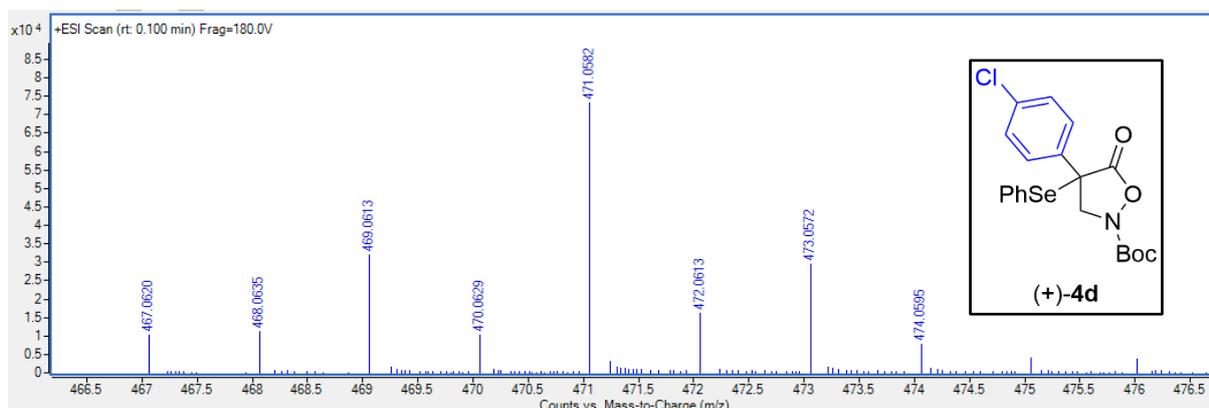
HRMS spectrum of 4b (ESI-QTOF, MeOH) m/z : $[M+NH_4]^+$ calculated for $C_{24}H_{27}N_2O_4Se^+$, 487.1131; found, 487.1129.



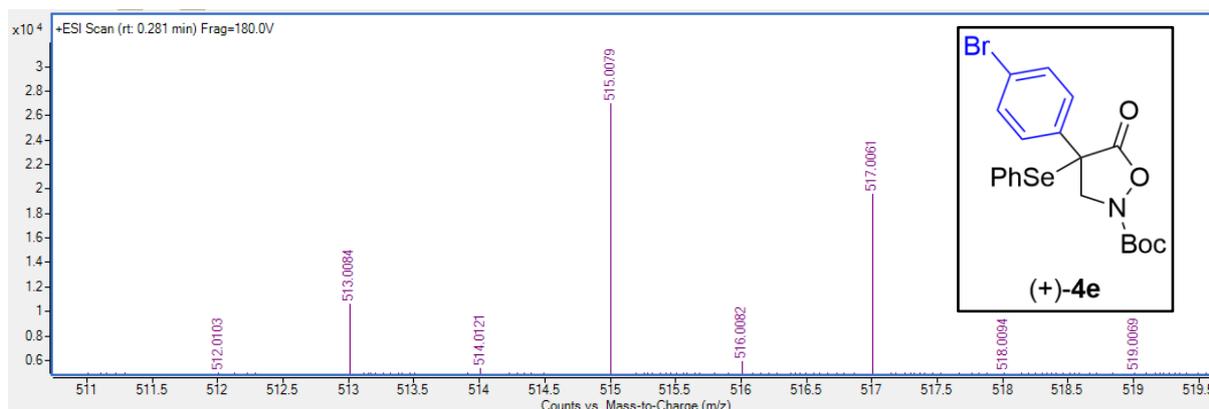
HRMS spectrum of 4c (ESI-QTOF, MeOH) m/z : $[M+NH_4]^+$ calculated for $C_{18}H_{23}N_2O_4Se^+$, 443.0538; found, 443.0538.



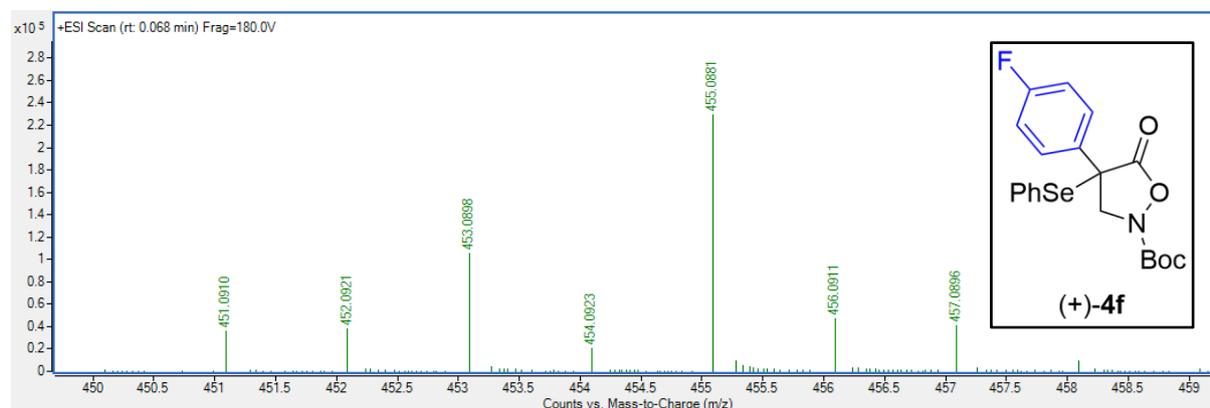
HRMS spectrum of 4d (ESI-QTOF, MeOH) m/z : $[M+NH_4]^+$ calculated for $C_{20}H_{24}ClN_2O_4Se^+$, 471.0584; found, 471.0582.



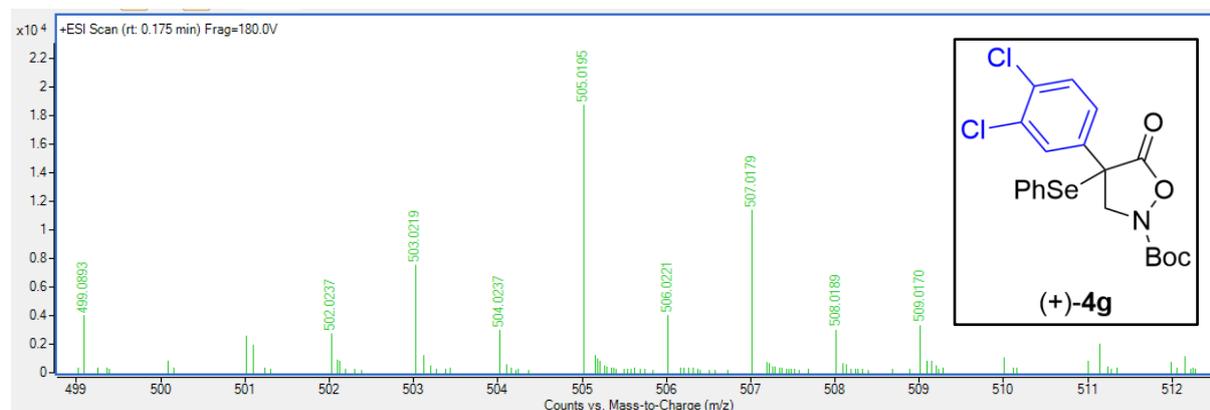
HRMS spectrum of 4e (ESI-QTOF, MeOH) m/z : $[M+NH_4]^+$ calculated for $C_{20}H_{24}BrN_2O_4Se^+$, 515.0079; found, 515.0079.



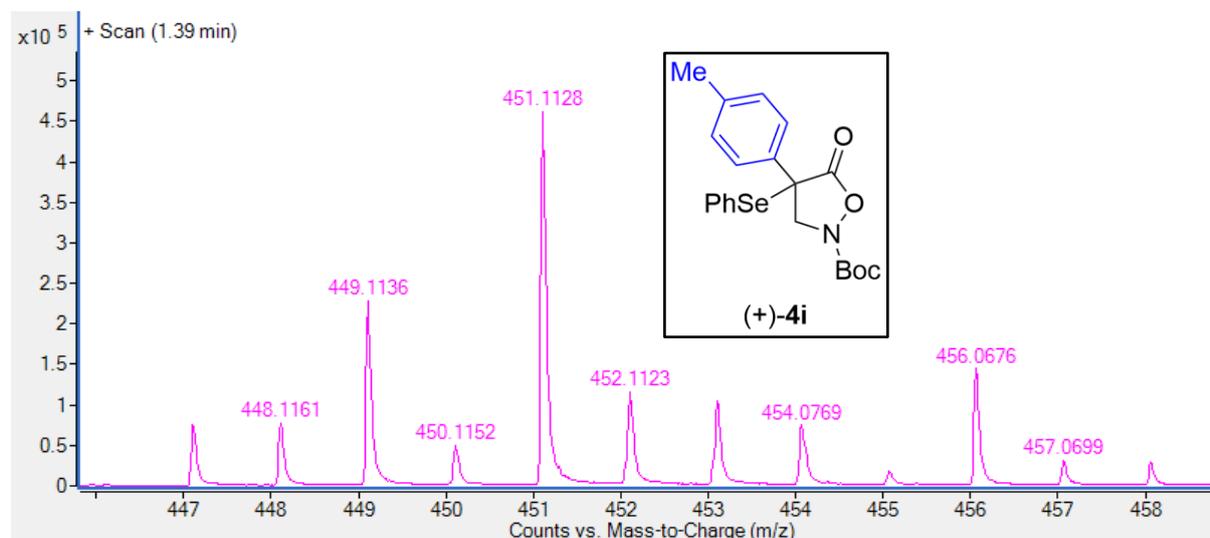
HRMS spectrum of 4f (ESI-QTOF, MeOH) m/z : $[M+NH_4]^+$ calculated for $C_{20}H_{24}FN_2O_4Se^+$, 455.0880; found, 455.0881.



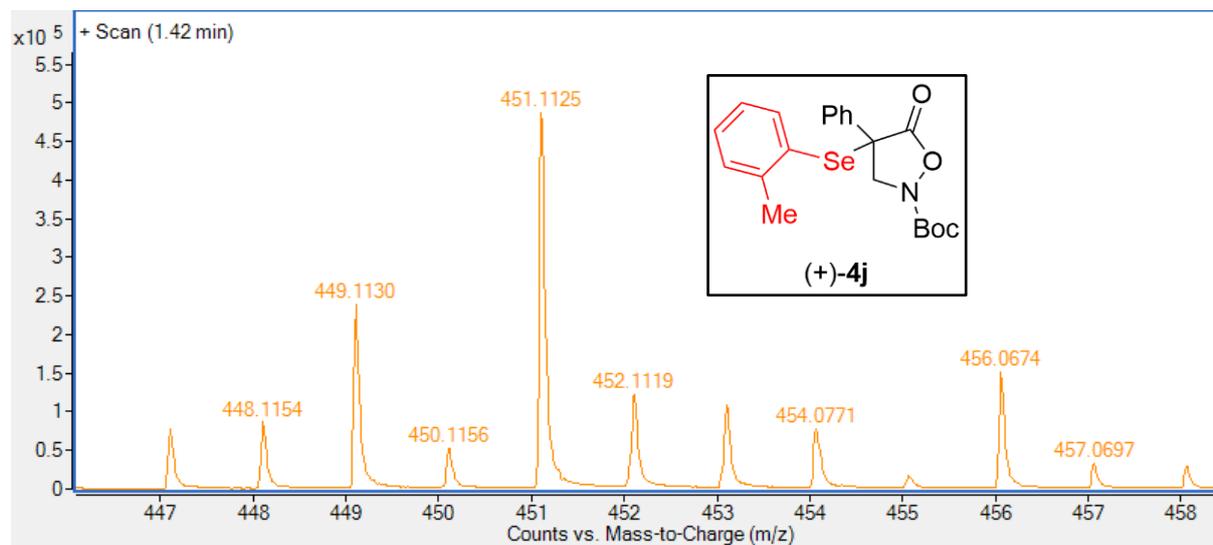
HRMS spectrum of 4g (ESI-QTOF, MeOH) m/z : $[M+NH_4]^+$ calculated for $C_{20}H_{23}Cl_2N_2O_4Se^+$, 505.0195; found, 505.0195.



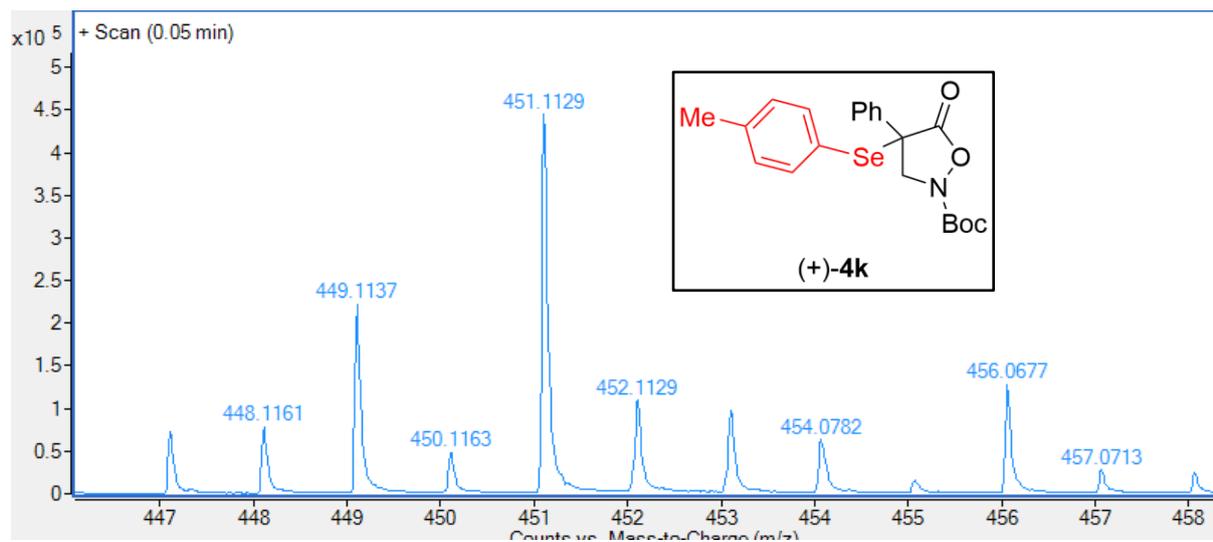
HRMS spectrum of 4i (ESI-QTOF, MeOH) m/z : $[M+NH_4]^+$ calculated for $C_{21}H_{27}N_2O_4Se^+$, 451.1131; found, 451.1128.



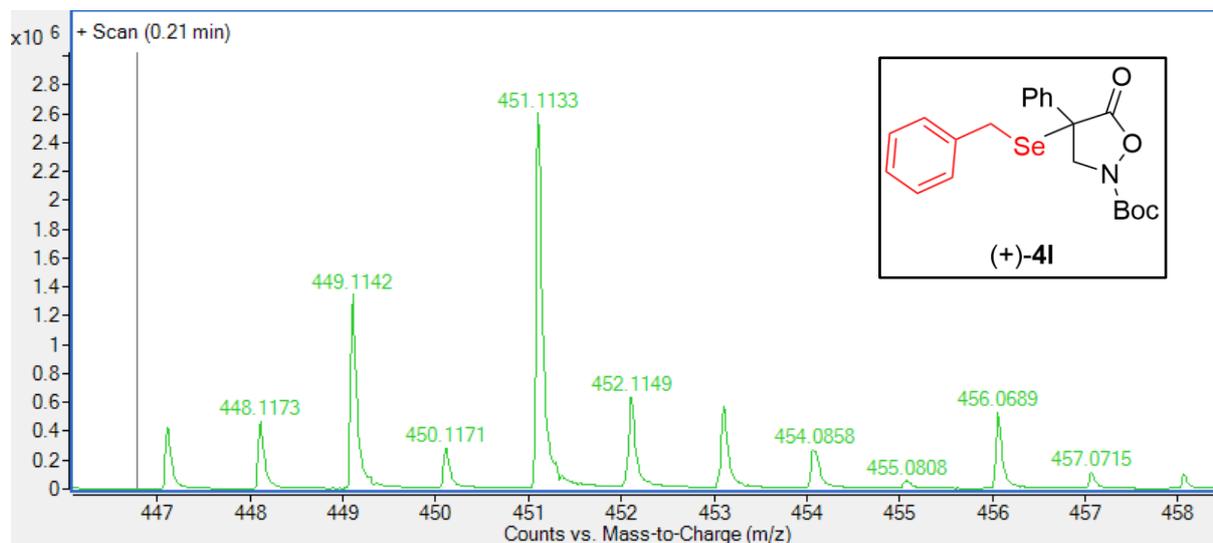
HRMS spectrum of 4j (ESI-QTOF, MeOH) m/z : $[M+NH_4]^+$ calculated for $C_{21}H_{27}N_2O_4Se^+$, 451.1131; found, 451.1125.



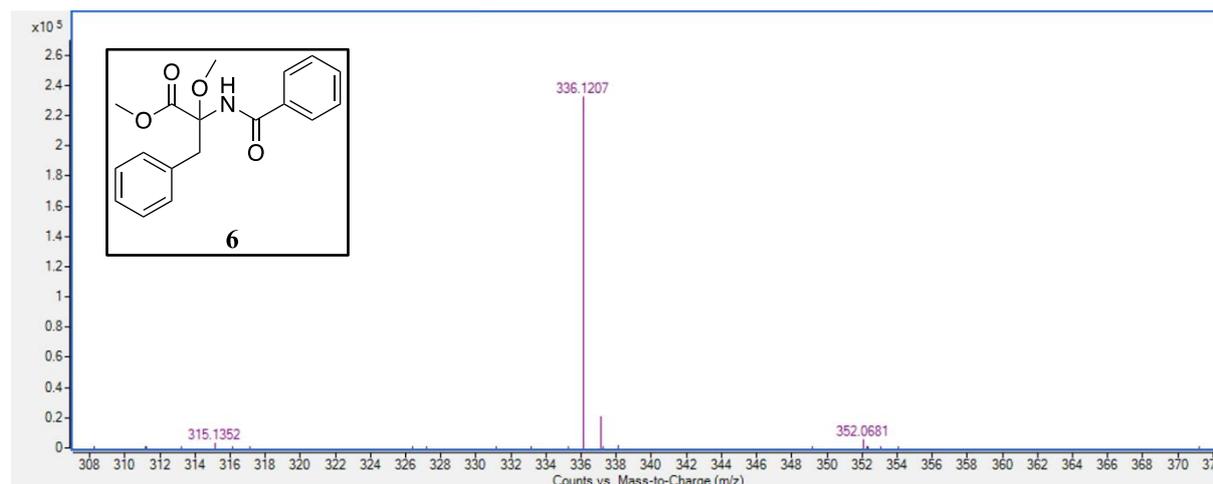
HRMS spectrum of 4k (ESI-QTOF, MeOH) m/z : $[M+NH_4]^+$ calculated for $C_{21}H_{27}N_2O_4Se^+$, 451.1131; found, 451.1129.



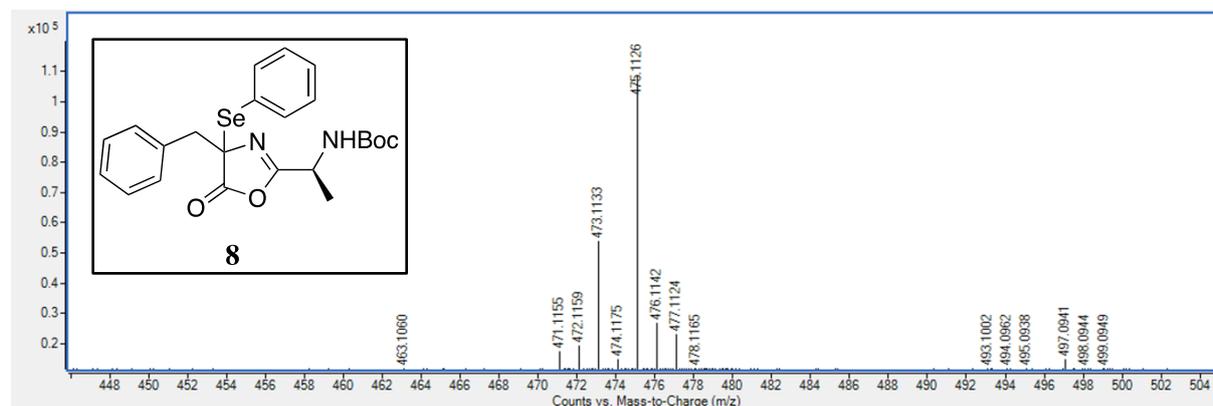
HRMS spectrum of 4I (ESI-QTOF, MeOH) m/z : $[M+NH_4]^+$ calculated for $C_{21}H_{27}N_2O_4Se^+$, 451.1131; found, 451.1133.



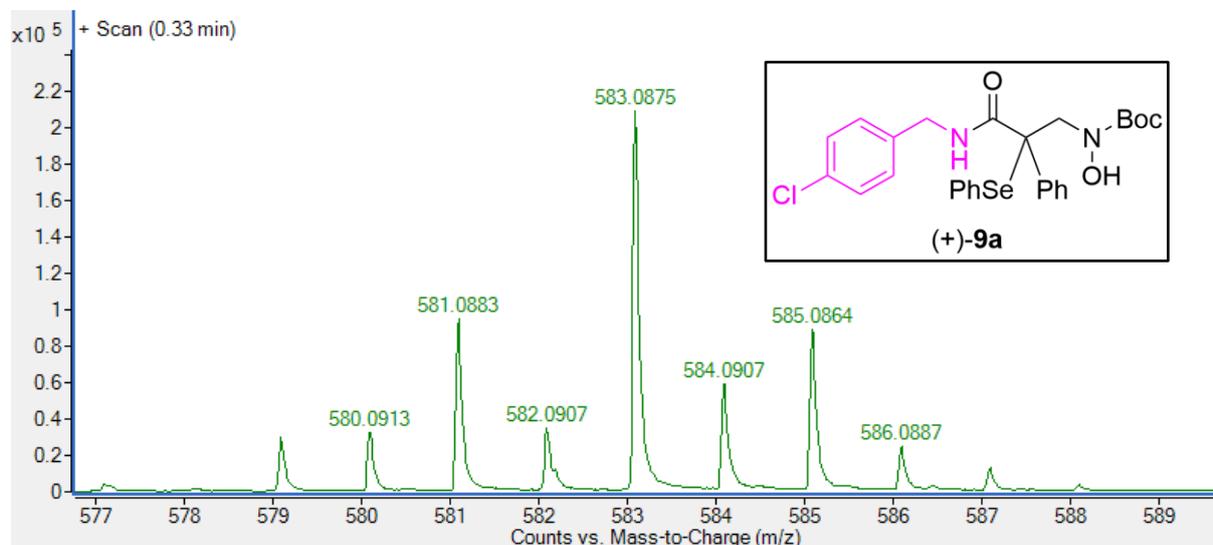
HRMS spectrum of 6 (ESI-QTOF, MeOH) m/z : $[M+Na]^+$ calculated for $C_{18}H_{19}NO_4Na$, 336.1206; found, 336.1207.



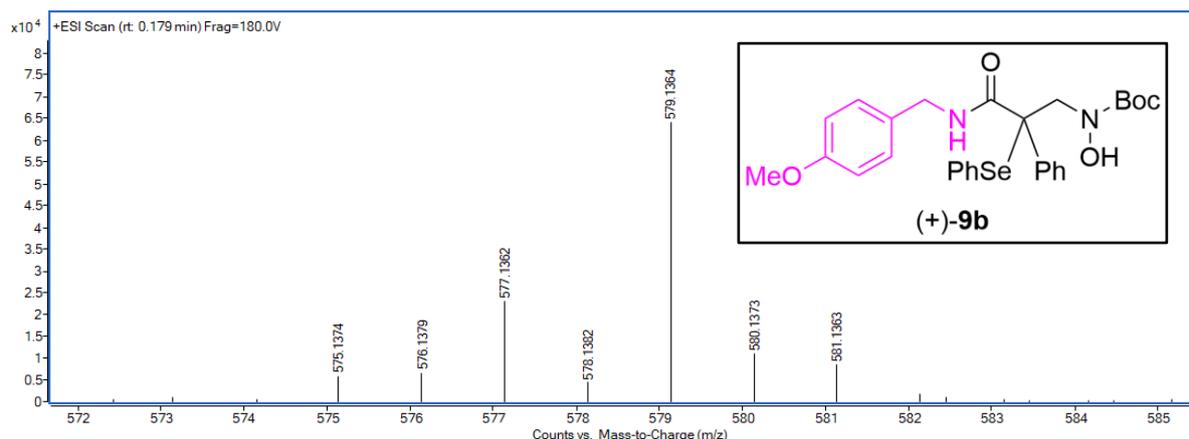
HRMS spectrum of 8 (ESI-QTOF, MeOH) m/z : $[M+H]^+$ calculated for $C_{23}H_{27}N_2O_4Se$, 475.1131; found, 475.1126.



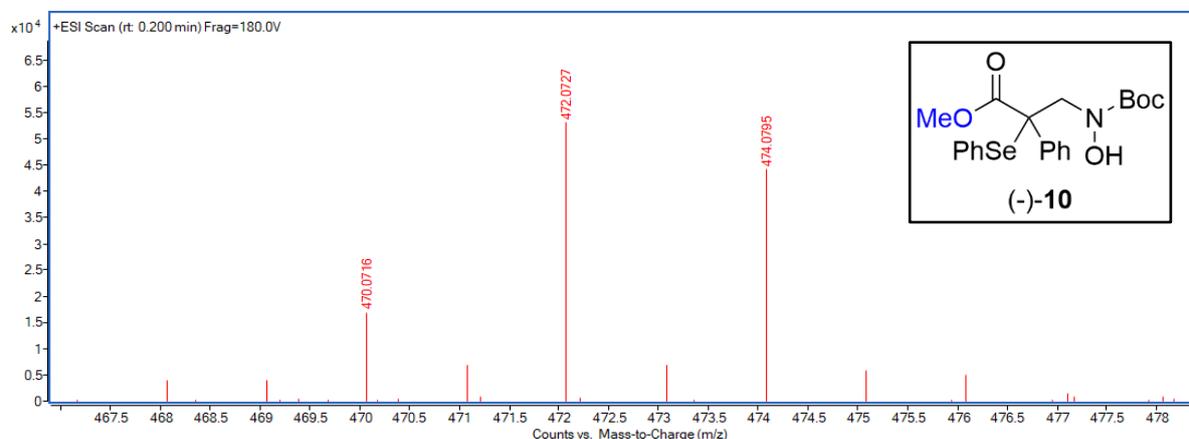
HRMS spectrum of 9a (ESI-QTOF, MeOH) m/z : $[M+Na]^+$ calculated for $C_{27}H_{29}ClN_2NaO_4Se^+$, 583.0873; found, 583.0875.



HRMS spectrum of 9b (ESI-QTOF, MeOH) m/z : $[M+Na]^+$ calculated for $C_{28}H_{32}N_2NaO_5Se^+$, 579.1369; found, 579.1364.



HRMS spectrum of 10 (ESI-QTOF, MeOH) m/z : $[M+Na]^+$ calculated for $C_{21}H_{25}NNaO_5Se^+$, 474.0790; found, 474.0795.



HRMS spectrum of 11 (ESI-QTOF, MeOH) m/z : $[M+Na]^+$ calculated for $C_{14}H_{15}NNaO_4^+$, 284.0893; found, 284.0893.

