Supporting Information

Preparation of Heterocycles via Visible-Light-Driven Aerobic Selenation of Olefins with Diselenides

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1. General Considerations

Unless otherwise stated, all reactions were set up in a clear pyrex glass tube and were stirred with a Teflon-coated magnetic stir bar. Analytical grade solvents and commercially available reagents were used to conduct the reactions. Reactions were monitored by thin layer chromatography (TLC), and the products were obtained by column chromatography on silica gel (200-300 mesh). NMR spectra were recorded on Bruker AM-400 MHz spectrometer for proton and carbon magnetic resonance spectra ($^1$H NMR and $^{13}$C NMR) in the solvent of CDCl$_3$ as the internal standard ($^1$H NMR: TMS at 0.00 ppm, CDCl$_3$ at 7.27 ppm; $^{13}$C NMR: CDCl$_3$ at 77.16 ppm. In reporting spectral data the format ($\delta$) chemical shift (multiplicity, $J$ values in Hz, integration) was used with the following abbreviations: s = singlet, d = doublet, t = triplet, q = quartet, quint = quintuplet, m = multiplet. Mass spectra were reported in units of m/z, and were obtained with esquire6000, TRACE DSQ and ORBITRAP ELITE. UV-Vis spectra and luminescence spectra were recorded on UV-2600, RF-5301PC respectively. Infrared spectra (IR) were recorded on a Nicolet (NEXUS 670).

2. Experimental Procedures

2.1 General Procedure for the Preparation of N-allylamides$^1$

To a solution of benzoyl chloride (1.45 mL, 12.5 mmol) in CH$_2$Cl$_2$ (20 mL) was added dropwise a solution of allylamine (0.75 mL, 10.0 mmol) and Et$_3$N (1.39 mL, 10.0 mmol) in CH$_2$Cl$_2$ (20 mL) at 0 °C. After the addition, the reaction was allowed to warm to room temperature and stirred overnight. The reaction was quenched with water (50 mL) and the aqueous layer was washed with CH$_2$Cl$_2$ (50 mL). The organic layers were combined and evaporated under vacuum. The residue was subjected to column chromatography on silica-gel to afford 1c.
2.2 The Structure of Substrates 4

![Substrates Diagram]

2.3 General Procedure for Seleno Cyclization of Olefins

A 10 mL a clear pyrex glass tube and was stirred with a Teflon-coated magnetic stir bar. Diselenide derivatives (0.06 mmol) and 4CzIPN (2 mol %) were added to the solution of olefins (0.1 mmol) in MeCN. Under visible-light generated from 3W blue LEDs, the reaction mixture was stirred at rt in the air. The reaction was monitored by TLC, after completion of the olefins, the solvent was removed under reduced pressure by rotary evaporator. Then, the residue was purified by silica gel column chromatography to give the desired product.
2.4 Gram-Scale Seleno Cyclization of 1i and Derivatization of 3ia

A 100 mL clear round bottom flask and was stirred with a Teflon-coated magnetic stir bar. Diphenyl diselenide (3.0 mmol) and 4CzIPN (0.5 mol %) were added to the solution of N-methylindole (5 mmol) in MeCN. The reaction mixture was stirred at rt 60 h, the solvent was removed under reduced pressure by rotary evaporator. Then, the residue was purified by silica gel column chromatography to give the desired product.

A mixture of Oxazoline 3ia (0.1 mmol) and 50% H$_2$O$_2$ (2 d) in THF (2 mL) was stirred at room temperature until complete consumption of the starting material, as monitored by TLC. After the evaporation of the solvent, the residual crude product was purified by flash chromatography with CH$_2$Cl$_2$/CH$_3$OH (v/v = 10:1).

Oxazoline 3ia (0.1 mmol) was dissolved in THF (2 mL) and 2M HCl (250 µL) was added. The reaction was allowed to stir at room temperature. The reaction was monitored by TLC, after completion of Oxazoline 3ia. 6M NaOH was added to adjusting the PH of reaction system and allowed stir at room temperature for 1 hr. The solvent was removed under reduced pressure by rotary evaporator. Then, the residue was purified by silica gel column chromatography to give the desired product.
3. Mechanism Studies

3.1. UV-Vis Absorption Spectra

![UV-Vis absorption spectra](image)

**Figure S1.** UV-Vis absorption spectra of 1a: N-allyl-1-naphthamide, 2a: Diphenyl diselenide and PC: 4CzIPN in MeCN.

3.2. Emission Quenching Experiments for PC

Emission intensities were recorded using a RF-5301PC Fluorescence Spectrometer. In a typical experiment, the emission spectrum of a $1 \times 10^{-5}$ M solution of PC in CH$_3$CN was collected. Then, appropriate amount of quencher was added to the measured solution and the emission spectrum of the sample was collected. Here $I_0$ and $I$ represent the intensities of the emission in the absence and presence of the quencher.

![Emission quenching](image)

**Figure S2.** The Emission Quenching of FIrPic by 1a and 2a
Figure S3. Stern–Volmer plots.

Table 1. Comparison of the Visible-Light Driven Aerobic Selenation with Previous Methods

| Entry | Substrates | Product | Yielda (%) | Yieldb (%) | Literature |
|-------|------------|---------|------------|------------|------------|
| 1     | 4e+2a      | 5ea     | 98         | 55         | J. Org. Chem. 1992, 57, 4019-4023 |
| 2     | 4f+2a      | 5fa     | 85         | 60         | J. Org. Chem. 1992, 57, 4019-4023 |
| 3     | 1c+2a      | 3ca     | 86         | 65         | Synth. Commun. 1998, 28, 1769-1778 |

a: Yield under standard conditions; b: Yield from the literatures.

4. Characterization Data

4.1. Characterization Data of Starting Materials

\[
\text{N-allyl-1-naphthamide (1a)}
\]

White solid, eluent (10% - 20% ethyl acetate in petroleum ether). $^1\text{H NMR (CDCl}_3, 400 \text{ MHz}) \delta 8.28-8.26 (m, 1H), 7.89-7.83 (m, 2H), 7.56-7.48 (m, 3H), 7.43-7.37 (m, 1H), 6.24 (brs, 1H), 5.97-5.89 (m, 1H), 5.19 (dd, $J = 17.2 \text{ Hz, } 10.4 \text{ Hz, } 2H), 4.10 (dd, J = 4.4 \text{ Hz, } 4.4 \text{ Hz, } 2H). \quad ^{13}\text{C NMR (CDCl}$_3$, 100 MHz) \delta 169.6, 134.5, 134.2, 133.8, 130.8, 130.3, 128.5, 127.3, 126.6, 125.5, 125.0, 124.8, 116.9, 42.5. \quad \text{EI-MS: m/z [M+H]'} 212.0
N-(2-methylallyl)-1-naphthamide (1b):
White solid, eluent (10% - 20% ethyl acetate in petroleum ether). $^1$H NMR (CDCl$_3$, 400 MHz) $\delta$ 8.29 (d, $J = 7.6$ Hz, 1H), 7.89-7.85 (m, 2H), 7.56-7.51 (m, 3H), 7.41-7.37 (m, 1H), 6.33 (brs, 1H), 4.92 (d, $J = 14.8$ Hz, 2H), 4.03 (s, 2H), 1.81 (s, 3H). $^{13}$C NMR (CDCl$_3$, 100 MHz) $\delta$ 169.6, 142.0, 134.6, 133.8, 130.7, 130.3, 128.4, 127.2, 126.5, 125.6, 124.9, 124.8, 111.3, 45.5, 20.6. EI-MS: m/z [M+H]$^+$ 226.1.

N-allylbenzamide (1c)
Colorless liquid, eluent (10% - 5% ethyl acetate in petroleum ether). $^1$H NMR (CDCl$_3$, 400 MHz) $\delta$ 7.81-7.79 (m, 2H), 7.46-7.43 (m, 1H), 7.37-7.34 (m, 2H), 7.03 (brs, 1H), 5.92-5.83 (m, 1H), 5.17 (dd, $J = 17.2$ Hz, 10.0 Hz, 2H), 4.01 (dd, $J = 5.6$ Hz, 1.6 Hz, 2H). $^{13}$C NMR (CDCl$_3$, 100 MHz) $\delta$ 167.7, 134.5, 134.3, 131.5, 128.5, 127.1, 116.4, 42.5. EI-MS: m/z [M+H]$^+$ 162.0.

N-allyl-4-methylbenzamide (1d):
White solid, eluent (10% - 20% ethyl acetate in petroleum ether). $^1$H NMR (CDCl$_3$, 400 MHz) $\delta$ 7.71 (d, $J = 8.0$ Hz, 2H), 7.22 (d, $J = 8.0$ Hz, 2H), 6.51 (brs, 1H), 5.97-5.87 (m, 1H), 5.26 (dd, $J = 17.2$ Hz, 1H), 5.16 (dd, $J = 10.4$ Hz, 1H), 4.06 (d, $J = 1.6$ Hz, 2H), 2.38 (s, 3H). $^{13}$C NMR (CDCl$_3$, 100 MHz) $\delta$ 167.5, 142.0, 134.5, 131.7, 129.3, 127.1, 116.6, 42.5, 21.6. EI-MS: m/z [M+H]$^+$ 175.9.
N-allyl-4-methoxybenzamide (1e):
White solid, eluent (10% - 20% ethyl acetate in petroleum ether). $^1$H NMR (CDCl$_3$, 400 MHz) $\delta$ 7.78–7.75 (m, 2H), 6.90-6.87 (m, 2H), 6.51 (brs, 1H), 5.96-5.86 (m, 1H), 5.25-5.19 (m, 1H), 5.15-5.12 (m, 1H), 4.05-4.02 (m, 2H), 3.82 (s, 3H). $^{13}$C NMR (CDCl$_3$, 100 MHz) $\delta$ 167.1, 162.3, 134.6, 128.9, 126.9, 116.5, 113.8, 55.5, 42.5. EI-MS: m/z [M+H]$^+$ 191.9.

N-allyl-4-bromobenzamide (1f):
White solid, eluent (10% - 20% ethyl acetate in petroleum ether). $^1$H NMR (CDCl$_3$, 400 MHz) $\delta$ 7.66-7.64 (m, 2H), 7.56-7.52 (m, 2H), 6.47 (brs, 1H), 5.95-5.86 (m, 1H), 5.26-5.17 (m, 2H), 4.05 (s, 2H). $^{13}$C NMR (CDCl$_3$, 100 MHz) $\delta$ 166.6, 134.1, 133.4, 132.0, 128.8, 126.4, 117.0, 42.7. EI-MS: m/z [M+H]$^+$ 240.1.

N-allyl-4-chlorobenzamide (1g):
White solid, eluent (10% - 20% ethyl acetate in petroleum ether). $^1$H NMR (CDCl$_3$, 400 MHz) $\delta$ 7.73 (dd, $J$ = 1.6 Hz, 1.6 Hz, 2H), 7.38 (dd, $J$ = 2.0 Hz, 0.8 Hz, 2H), 6.57 (brs, 1H), 5.95-5.86 (m, 1H), 5.26 (d, $J$ = 17.2 Hz, 1H), 5.18 (d, $J$ = 10.4 Hz, 1H), 4.06-4.03 (m, 2H). $^{13}$C NMR (CDCl$_3$, 100 MHz) $\delta$ 166.6, 137.9, 134.1, 133.0, 128.9, 128.6, 116.9, 42.7. EI-MS: m/z [M+H]$^+$ 195.9.

N-allyl-4-fluorobenzamide (1h):
White solid, eluent (10% - 20% ethyl acetate in petroleum ether). $^1$H NMR (CDCl$_3$, 400 MHz) $\delta$ 7.82-7.79 (m, 2H), 7.09 (t, $J$ = 8.6 Hz, 2H), 6.44 (brs, 1H), 5.97-5.87 (m, 1H), 5.26 (dd, $J$ = 16.8 Hz, 1H), 5.18 (dd, $J$ = 10.4 Hz, 1H), 4.07-4.04 (m, 2H). $^{13}$C NMR (CDCl$_3$, 100 MHz) $\delta$ 166.5, 166.2 (d, $J$ = 251.0 Hz), 134.2, 130.8, 129.5 (d, $J$
= 9.0 Hz), 116.9, 115.9 (d, J = 22.0 Hz), 42.7. **EI-MS**: m/z [M+H]^+ 179.9.

\[ \begin{array}{c}
\text{F}_3\text{C} \\
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\text{CCH}_2
\end{array} \]

*N*-allyl-4-(trifluoromethyl)benzamide (1i)

White solid, eluent (10% - 20% ethyl acetate in petroleum ether). **\(^1\)H NMR (CDCl\textsubscript{3}, 400 MHz)** \(\delta\) 7.90 (d, \(J = 8.0\) Hz, 2H), 7.69 (d, \(J = 8.0\) Hz, 2H), 6.53 (brs, 1H), 5.98-5.88 (m, 1H), 5.28 (dd, \(J = 17.2\) Hz, 1H), 5.22-5.19 (m, 1H), 4.10-4.07 (m, 2H). **\(^{13}\)C NMR (CDCl\textsubscript{3}, 100 MHz)** \(\delta\) 166.4, 137.9, 133.9, 133.5 (d, \(J = 33.0\) Hz), 127.7, 125.8 (q, \(J = 3.3\) Hz), 125.2 (d, \(J = 271\) Hz), 117.1, 42.8. **EI-MS**: m/z [M+H]^+ 230.0.

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\text{C} \\
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\text{CCH}_2
\end{array} \]

*N*-allylthiophene-2-carboxamide (1j)

White solid, eluent (10% - 20% ethyl acetate in petroleum ether). **\(^1\)H NMR (CDCl\textsubscript{3}, 400 MHz)** \(\delta\) 7.59 (d, \(J = 2.8\) Hz, 1H), 7.46 (dd, \(J = 5.0\) Hz, 1H), 7.05 (t, \(J = 4.2\) Hz, 1H), 6.59 (brs, 1H), 5.95-5.85 (m, 1H), 5.21 (dd, \(J = 17.2\) Hz, 10.4 Hz, 2H), 4.05 (d, \(J = 4.0\) Hz, 2H). **\(^{13}\)C NMR (CDCl\textsubscript{3}, 100 MHz)** \(\delta\) 162.1, 139.1, 134.2, 130.1, 128.3, 127.8, 116.8, 42.5. **EI-MS**: m/z [M+H]^+ 168.0.

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\text{CCH}_2
\end{array} \]

*N*-allylfuran-2-carboxamide (1k)

White solid, eluent (10% - 20% ethyl acetate in petroleum ether). **\(^1\)H NMR (CDCl\textsubscript{3}, 400 MHz)** \(\delta\) 7.40 (d, \(J = 0.8\) Hz, 1H), 7.09 (d, \(J = 3.6\) Hz, 1H), 6.59 (brs, 1H), 6.46 (dd, \(J = 3.4\) Hz, 1H), 5.92-5.83 (m, 1H), 5.24 (dd, \(J = 17.2\) Hz, 1H), 5.15 (dd, \(J = 10.4\) Hz, 1H), 4.02 (m, \(J = 5.8\) Hz, 2H). **\(^{13}\)C NMR (CDCl\textsubscript{3}, 100 MHz)** \(\delta\) 158.4, 148.1, 144.0, 134.1, 116.7, 114.3, 112.2, 41.6. **EI-MS**: m/z [M+H]^+ 152.1.

\[ \begin{array}{c}
\text{C} \\
\text{N} \\
\text{CCH}_2
\end{array} \]

*N*-allyl-2-phenylacetamide (1l)

White solid, eluent (10% - 20% ethyl acetate in petroleum ether). **\(^1\)H NMR (CDCl\textsubscript{3}, 400 MHz)** \(\delta\) ...
400 MHz) δ 7.37-7.30 (m, 2H), 7.29-7.26 (m, 3H), 5.81-5.71 (m, 2H), 5.08-5.03 (m, 2H), 3.84-3.82 (m, 2H), 3.58 (s, 2H). 13C NMR (CDCl3, 100 MHz) δ 171.0, 135.0, 134.2, 129.6, 129.1, 127.5, 116.1, 43.8, 42.0. EI-MS: m/z [M+H]+ 176.0.

4.2. Characterization Data of Products

2-(naphthalen-1-yl)-5-((phenylselanyl)methyl)-4,5-dihydrooxazole (3aa): 34.5 mg (yield: 94%, 0.1mmol scale), colorless liquid, eluent (6% ethyl acetate in petroleum ether). 1H NMR (CDCl3, 400 MHz) δ 9.14 (d, J = 8.8 Hz, 1H), 7.97-7.92 (m, 2H), 7.87-7.85 (d, J = 8.0 Hz, 1H), 7.60-7.56 (m, 3H), 7.53-7.49 (m, 1H), 7.48-7.42 (m, 1H), 7.28-7.25 (m, 3H), 4.93-4.89 (m, 1H), 4.32 (dd, J = 15.0 Hz, 1H), 4.00 (dd, J = 15.2 Hz, 1H), 3.35 (dd, J = 12.6 Hz, 1H), 3.13 (dd, J = 12.6 Hz, 1H). 13C NMR (CDCl3, 100 MHz) δ 163.7, 133.9, 133.6, 132.2, 131.3, 129.5, 129.3, 129.1, 128.7, 127.7, 127.6, 126.6, 126.3, 124.8, 124.4, 77.9, 61.1, 32.2. IR νmax: 3444, 1643, 1511, 1477, 1401, 1216, 1191, 1123, 985, 906, 808, 777, 735. ESI-HRMS: m/z calculated for C20H17NOSe [M+H]+ 368.0548, found 368.0544.

5-methyl-2-(naphthalen-1-yl)-5-((phenylselanyl)methyl)-4,5-dihydrooxazole (3ba): 37.3 mg (yield: 97%, 0.1mmol scale), colorless liquid, eluent (6% ethyl acetate in petroleum ether). 1H NMR (CDCl3, 400 MHz) δ 9.12 (d, J = 8.6 Hz, 1H), 7.93-7.84 (m, 3H), 7.60-7.52 (m, 4H), 7.50 (t, J = 6.8 Hz, 1H), 7.48-7.19 (m, 3H), 4.18 (d, J = 14.8 Hz, 1H), 3.97 (d, J = 14.8 Hz, 1H), 3.35 (d, J = 3.2 Hz, 2H), 1.64 (s, 3H). 13C NMR (CDCl3, 100 MHz) δ 163.1, 133.9, 133.3, 132.0, 131.3, 130.6, 129.4, 129.2, 128.6, 127.5, 127.4, 126.6, 126.2, 124.8, 84.8, 66.4, 38.7, 26.2. IR νmax: 3443, 1641, 1511, 1330, 1254, 1124, 1072, 1009, 808, 777, 738. ESI-HRMS: m/z calculated for C21H19NOSe [M+H]+ 382.0705, found 382.0704.
2-phenyl-5-((phenylselanyl)methyl)-4,5-dihydrooxazole (3ca): 27.3 mg (yield: 86%, 0.1mmol scale), colorless liquid, eluent (6% ethyl acetate in petroleum ether). $^1$H NMR (CDCl$_3$, 400 MHz) $\delta$ 7.87-7.84 (m, 2H), 7.58-7.55 (m, 2H), 7.48-7.44 (m, 1H), 7.39-7.36 (m, 2H), 7.28-7.25 (m, 3H), 4.91-4.86 (m, 1H), 4.16 (dd, $J$ = 15.0 Hz, 1H), 3.83 (dd, $J$ = 14.8 Hz, 1H), 3.29 (dd, $J$ = 12.8 Hz, 1H), 3.05 (dd, $J$ = 12.8 Hz, 1H). $^{13}$C NMR (CDCl$_3$, 100 MHz) $\delta$ 163.9, 133.6, 131.5, 129.4, 129.1, 128.5, 128.3, 127.8, 127.7, 79.1, 60.4, 32.1. IR $\nu_{\text{max}}$: 3441, 1650, 1579, 1478, 1335, 1259, 1062, 1024, 1000, 780, 738, 693. ESI-MS: m/z [M+H]$^+$ 317.9.

5-((phenylselanyl)methyl)-2-(p-tolyl)-4,5-dihydrooxazole (3da): 27.9 mg (yield: 85%, 0.1mmol scale), colorless liquid, eluent (6% ethyl acetate in petroleum ether). $^1$H NMR (CDCl$_3$, 400 MHz) $\delta$ 7.75 (d, $J$ = 8.4 Hz, 2H), 7.57-7.55 (m, 2H), 7.28-7.25 (m, 3H), 7.19 (d, $J$ = 8.0 Hz, 2H), 4.90-4.83 (m, 1H), 4.14 (dd, $J$ = 14.8 Hz, 1H), 3.81 (dd, $J$ = 14.8 Hz, 1H), 3.29 (dd, $J$ = 12.8 Hz, 1H), 3.03 (dd, $J$ = 12.8 Hz, 1H), 2.38 (s, 3H). $^{13}$C NMR (CDCl$_3$, 100 MHz) $\delta$ 164.0, 141.9, 133.6, 129.4, 129.2, 128.3, 127.8, 127.7, 125.1, 79.0, 60.4, 32.2, 21.8. IR $\nu_{\text{max}}$: 3427, 1649, 1478, 1335, 1260, 1070, 1021, 829, 738, 691. ESI-HRMS: m/z calculated for C$_{17}$H$_{17}$NOSe [M+H]$^+$ 332.0548, found 332.0547.

2-(4-methoxyphenyl)-5-((phenylselanyl)methyl)-4,5-dihydrooxazole (3ea): 29.4 mg (yield: 85%, 0.1mmol scale), colorless liquid, eluent (6% ethyl acetate in petroleum ether). $^1$H NMR (CDCl$_3$, 400 MHz) $\delta$ 7.81-7.78 (m, 2H), 7.57-7.55 (m, 2H), 7.27-7.24 (m, 3H), 6.89-6.84 (m, 2H), 4.89-4.84 (m, 1H), 4.13 (dd, $J$ = 14.8 Hz, 1H), 3.83 (s, 3H), 3.80 (dd, $J$ = 14.8 Hz, 1H), 3.28 (dd, $J$ = 12.8 Hz, 1H), 3.04 (dd, $J$ = 12.8 Hz, 1H). $^{13}$C NMR (CDCl$_3$, 100 MHz) $\delta$ 163.7, 162.3, 133.5, 130.1, 129.4, 129.2, 127.6, 120.4, 113.8, 79.0, 60.4, 55.5, 32.2. IR $\nu_{\text{max}}$: 3434, 2360, 2342, 1648, 1609,
1513, 1335, 1257, 1171, 1071, 1027, 946, 841, 739. **ESI-HRMS:** m/z calculated for C_{17}H_{17}NO_{2}Se [M+H]^+ 348.0497, found 348.0496.

![](image_url)

2-(4-bromophenyl)-5-((phenylselanyl)methyl)-4,5-dihydrooxazole (3fa): 29.5 mg (yield: 74%, 0.1mmol scale), colorless liquid, eluent (6% ethyl acetate in petroleum ether). **^1H NMR (CDCl₃, 400 MHz)** δ 7.71-7.67 (m, 2H), 7.57-7.54 (m, 2H), 7.52-7.49 (m, 2H), 7.28-7.24 (m, 3H), 4.93-4.87 (m, 1H), 4.14 (dd, J = 15.2 Hz, 1H), 3.81 (dd, J = 15.2 Hz, 1H), 3.27 (dd, J = 12.8 Hz, 1H), 3.06 (dd, J = 12.8 Hz, 1H). **^{13}C NMR (CDCl₃, 100 MHz)** δ 163.2, 133.6, 131.8, 129.9, 129.5, 129.1, 127.8, 126.8, 126.2, 79.4, 60.5, 32.1. **IR ν max:** 3444, 1650, 1486, 1399, 1334, 1259, 1072, 1012, 837, 738, 691. **ESI-HRMS:** m/z calculated for C_{16}H_{14}BrNOSe [M+H]^+ 395.9497, found 395.9492.

![](image_url)

2-(4-chlorophenyl)-5-((phenylselanyl)methyl)-4,5-dihydrooxazole (3ga): 31.2 mg (yield: 89%, 0.1mmol scale), colorless liquid, eluent (6% ethyl acetate in petroleum ether). **^1H NMR (CDCl₃, 400 MHz)** δ 7.77-7.75 (m, 2H), 7.58-7.53 (m, 2H), 7.35-7.33 (m, 2H), 7.28-7.24 (m, 3H), 4.93-4.86 (m, 1H), 4.14 (dd, J = 15.2 Hz, 1H), 3.82 (dd, J = 15.2 Hz, 1H), 3.27 (dd, J = 12.8 Hz, 1H), 3.06 (dd, J = 12.8 Hz, 1H). **^{13}C NMR (CDCl₃, 100 MHz)** δ 163.1, 137.7, 133.6, 129.7, 129.5, 129.1, 128.8, 127.8, 126.4, 79.4, 60.5, 32.1. **IR ν max:** 3439, 1651, 1490, 1404, 1334, 1259, 1091, 1069, 1015, 840, 736, 691. **ESI-HRMS:** m/z calculated for C_{16}H_{14}ClNOSe [M+H]^+ 352.0002, found 352.0000.

![](image_url)

2-(4-fluorophenyl)-5-((phenylselanyl)methyl)-4,5-dihydrooxazole (3ha): 30.9 mg (yield: 92%, 0.1mmol scale), colorless liquid, eluent (6% ethyl acetate in petroleum ether). **^1H NMR (CDCl₃, 400 MHz)** δ 7.83-7.82 (m, 2H), 7.58-7.55 (m, 2H), 7.28-7.24 (m, 2H), 7.08-7.03 (m, 3H), 4.92-4.85 (m, 1H), 4.15 (dd, J = 15.2 Hz, 1H),
3.82 (dd, J = 15.2 Hz, 1H), 3.27 (dd, J = 12.8 Hz, 1H), 3.06 (dd, J = 12.6 Hz, 1H). $^{13}$C NMR (CDCl$_3$, 100 MHz) δ 166.1 (d, J = 251 Hz), 163.0, 133.6, 130.6 (d, J = 9 Hz), 129.4, 129.1, 127.7, 124.1, 115.7 (d, J = 21 Hz), 79.3, 60.5, 32.1. IR $v_{\text{max}}$: 3439, 1651, 1606, 1509, 1335, 1259, 1225, 1154, 1069, 846, 736, 691. ESI-HRMS: m/z calculated for C$_{16}$H$_{14}$FNOSe $[M+H]^+$ 336.0297, found 336.0297.

5-((phenylselanyl)methyl)-2-(4-(trifluoromethyl)phenyl)-4,5-dihydrooxazole (3ia): 36.1 mg (yield: 93%, 0.1mmol scale), colorless liquid, eluent (6% ethyl acetate in petroleum ether). $^1$H NMR (CDCl$_3$, 400 MHz) δ 7.95 (d, J = 8.0 Hz, 2H), 7.64 (d, J = 8.4 Hz, 2H), 7.58-7.55 (m, 2H), 7.28-7.24 (m, 3H), 4.97-4.90 (m, 1H), 4.19 (dd, J = 15.2 Hz, 1H), 3.87 (dd, J = 15.2 Hz, 1H), 3.28 (dd, J = 12.8 Hz, 1H), 3.09 (dd, J = 13.0 Hz, 1H). $^{13}$C NMR (CDCl$_3$, 100 MHz) δ 162.7, 133.6, 133.3 (d, J = 32.0 Hz), 131.2 (d, J = 217.0 Hz), 129.5, 128.7, 127.8, 125.5 (q, J = 3.3 Hz), 125.3 (d, J = 271.0 Hz), 79.5, 60.3, 31.8. IR $v_{\text{max}}$: 3411, 1652, 1413, 1322, 1167, 1126, 1074, 1018, 961, 853, 738, 673. ESI-HRMS: m/z calculated for C$_{17}$H$_{14}$F$_3$NOSSe $[M+H]^+$ 386.0265, found 386.0262.

5-((phenylselanyl)methyl)-2-(thiophen-2-yl)-4,5-dihydrooxazole (3ja): 29.0 mg (yield: 90%, 0.1mmol scale), colorless liquid, eluent (6% ethyl acetate in petroleum ether). $^1$H NMR (CDCl$_3$, 400 MHz) δ 7.57-7.54 (m, 2H), 7.50-7.48 (m, 1H), 7.44-7.42 (m, 1H), 7.28-7.25 (m, 3H), 7.05 (dd, J = 5.0 Hz, 1H), 4.91-4.84 (m, 1H), 4.14 (dd, J = 14.8 Hz, 1H), 3.81 (dd, J = 14.8 Hz, 1H), 3.29 (dd, J = 12.4 Hz, 1H), 3.04 (dd, J = 12.8 Hz, 1H). $^{13}$C NMR (CDCl$_3$, 100 MHz) δ 159.7, 133.6, 130.5, 130.0, 129.4, 129.0, 127.8, 127.7, 79.6, 60.4, 31.9. IR $v_{\text{max}}$: 3432, 1649, 1477, 1434, 1331, 1252, 1058, 1020, 737, 716, 691. ESI-HRMS: m/z calculated for C$_{14}$H$_{13}$NOSSe $[M+H]^+$ 323.9956, found 323.9955.
2-(furan-2-yl)-5-((phenylselanyl)methyl)-4,5-dihydrooxazole (3ka): 13.5 mg (yield: 44%, 0.1mmol scale), colorless liquid, eluent (6% ethyl acetate in petroleum ether).

$^1$H NMR (CDCl$_3$, 400 MHz) $\delta$ 7.57-7.52 (m, 3H), 7.29-7.26 (m, 3H), 6.87 (d, $J$ = 3.2 Hz, 1H), 6.47 (dd, $J$ = 3.6 Hz, 1H), 4.89-4.82 (m, 1H), 4.16 (dd, $J$ = 15.0 Hz, 1H), 3.84 (dd, $J$ = 14.8 Hz, 1H), 3.29 (dd, $J$ = 12.8 Hz, 1H), 3.03 (dd, $J$ = 12.8 Hz, 1H).

$^{13}$C NMR (CDCl$_3$, 100 MHz) $\delta$ 156.3, 145.4, 143.1, 133.6, 129.5, 128.9, 127.8, 114.6, 111.7, 79.4, 60.3, 31.8. IR $\nu_{\text{max}}$: 3440, 1653, 1479, 1331, 1265, 1169, 1091, 1011, 937,740, 691. ESI-HRMS: m/z calculated for C$_{14}$H$_{13}$NO$_2$Se [M+H]$^+$ 308.0184, found 308.0183.

![Structure](image)

phenyl(5-((phenylselanyl)methyl)-4,5-dihydrooxazol-2-yl)methanone (3la): 18.3 mg (yield: 53%, 0.1mmol scale), colorless liquid, eluent (6% ethyl acetate in petroleum ether).

$^1$H NMR (CDCl$_3$, 400 MHz) $\delta$ 7.94 (d, $J$ = 8.0 Hz, 2H), 7.58-7.56 (m, 2H), 7.47 (t, $J$ = 7.0 Hz, 1H), 7.41 (t, $J$ = 7.6 Hz, 2H), 7.30 (t, $J$ = 2.6 Hz, 3H), 4.66-4.59 (m, 1H), 4.39 (dd, $J$ = 17.2 Hz, 1H), 3.72 (dd, $J$ = 17.2 Hz, 1H), 3.29 (dd, $J$ = 13.4 Hz, 1H), 3.12 (dd, $J$ = 13.2 Hz, 1H).

$^{13}$C NMR (CDCl$_3$, 100 MHz) $\delta$ 159.2, 155.4, 134.0, 133.7, 131.4, 129.7, 128.9, 128.8, 128.5, 128.1, 77.0, 52.3, 28.6. IR $\nu_{\text{max}}$: 3057, 1735, 1611, 1578, 1478, 1437, 1176, 1051, 1022, 934, 739, 691. ESI-HRMS: m/z calculated for C$_{17}$H$_{15}$NO$_2$Se [M+H]$^+$ 346.0341, found 346.0341.

![Structure](image)

2-(naphthalen-1-yl)-5-((p-tolylselanyl)methyl)-4,5-dihydrooxazole (3ab): 30.6 mg (yield: 80%, 0.1mmol scale), colorless liquid, eluent (6% ethyl acetate in petroleum ether).

$^1$H NMR (CDCl$_3$, 400 MHz) $\delta$ 9.14 (d, $J$ = 8.4 Hz, 1H), 7.94 (d, $J$ = 8.0 Hz, 2H), 7.86 (d, $J$ = 8.0 Hz, 1H), 7.60-7.55 (m, 1H), 7.52-7.47 (m, 1H), 7.45-7.41 (m, 1H), 7.08 (d, $J$ = 8.0 Hz, 2H), 4.92-4.84 (m, 1H), 4.30 (dd, $J$ = 15.2 Hz, 1H), 3.98 (dd, $J$ = 15.2 Hz, 1H), 3.30 (dd, $J$ = 12.6 Hz, 1H), 3.07 (dd, $J$ = 12.8 Hz, 1H), 2.31 (s, 3H).
$^{13}$C NMR (CDCl$_3$, 100 MHz) $\delta$ 163.7, 137.9, 134.2, 133.9, 132.1, 131.3, 130.3, 129.3, 128.6, 127.5, 126.6, 126.3, 125.2, 124.8, 124.6, 78.0, 61.2, 32.5, 21.3. IR $\nu_{\text{max}}$: 3446, 1642, 1590, 1511, 1489, 1318, 1244, 1191, 1122, 984, 805, 777. ESI-HRMS: m/z calculated for C$_{21}$H$_{19}$NOSe [M+H]$^+$ 382.0705, found 382.0703.

5-(((4-methoxyphenyl)selanyl)methyl)-2-(naphthalen-1-yl)-4,5-dihydrooxazole (3ac):
31.0 mg (yield: 78%, 0.1mmol scale), colorless liquid, eluent (6% ethyl acetate in petroleum ether). $^1$H NMR (CDCl$_3$, 400 MHz) $\delta$ 9.13 (d, $J = 8.4$ Hz, 1H), 7.96-7.92 (m, 2H), 7.86 (d, $J = 8.0$ Hz, 1H), 7.60-7.52 (m, 4H), 7.50-7.42 (m, 1H), 6.81-6.78 (m, 2H), 4.89-4.84 (m, 1H), 4.30 (dd, $J = 14.8$ Hz, 1H), 3.97 (dd, $J = 14.8$ Hz, 1H), 3.77 (s, 3H). $^{13}$C NMR (CDCl$_3$, 100 MHz) $\delta$ 163.7, 159.9, 136.5, 133.9, 132.1, 131.3, 129.3, 128.6, 127.5, 126.6, 126.3, 124.8, 124.6, 118.9, 115.2, 78.0, 61.1, 55.5, 33.1. IR $\nu_{\text{max}}$: 3432, 1642, 1590, 1490, 1318, 1285, 1246, 1174, 1122, 1028, 983, 808, 777. ESI-HRMS: m/z calculated for C$_{21}$H$_{19}$NO$_2$Se [M+H]$^+$ 398.0654, found 398.0651.

5-(((2-methoxyphenyl)selanyl)methyl)-2-(naphthalen-1-yl)-4,5-dihydrooxazole (3ad):
29.8 mg (yield: 75%, 0.1mmol scale), colorless liquid, eluent (6% ethyl acetate in petroleum ether). $^1$H NMR (CDCl$_3$, 400 MHz) $\delta$ 9.15 (d, $J = 8.8$ Hz, 1H), 8.00-7.98 (m, 1H), 7.94 (d, $J = 8.4$ Hz, 1H), 7.86 (d, $J = 8.0$ Hz, 1H), 7.60-7.56 (m, 1H), 7.52-7.48 (m, 2H), 7.44 (t, $J = 8.0$ Hz, 1H), 7.26-7.23 (m, 1H), 6.91-6.83 (m, 2H), 4.96-4.89 (m, 1H), 4.32 (dd, $J = 15.2$ Hz, 1H), 4.03 (dd, $J = 15.2$ Hz, 1H), 3.87 (s, 3H), 3.38 (dd, $J = 12.4$ Hz, 1H), 3.09 (dd, $J = 12.8$ Hz, 1H). $^{13}$C NMR (CDCl$_3$, 100 MHz) $\delta$ 163.7, 158.5, 133.9, 132.9, 132.1, 131.3, 129.3, 128.9, 128.6, 127.5, 126.6, 126.3, 124.8, 124.6, 121.7, 118.1, 110.9, 77.9, 71.3, 56.1, 29.6. IR $\nu_{\text{max}}$: 3447, 1642, 1512, 1474, 1318, 1244, 1123, 1024, 984, 808, 778, 749. ESI-HRMS: m/z calculated
for C_{21}H_{19}NO_2Se [M+H]^+ 398.0654, found 398.0653.

2-(naphthalen-1-yl)-5-(((3,4,5-trimethoxyphenyl)selanyl)methyl)-4,5-dihydrooxazole (3ae): 20.6 mg (yield: 45%, 0.1mmol scale), colorless liquid, eluent (6% ethyl acetate in petroleum ether). ¹H NMR (CDCl₃, 400 MHz) δ 9.15 (d, J = 8.8 Hz, 1H), 7.96 (t, J = 8.0 Hz, 2H), 7.88 (d, J = 8.0 Hz, 1H), 7.62-7.59 (m, 1H), 7.58 (d, J = 1.2 Hz, 1H), 7.53-7.44 (m, 1H), 6.83 (s, 2H), 5.00-4.93 (m, 1H), 4.36 (dd, J = 15.2 Hz, 1H), 4.03 (dd, J = 15.2 Hz, 1H), 3.83 (s, 3H), 3.79 (s, 6H), 3.34 (dd, J = 12.4 Hz, 1H), 3.17 (dd, J = 12.4 Hz, 1H). ¹³C NMR (CDCl₃, 100 MHz) δ 163.7, 153.6, 133.9, 132.2, 131.3, 129.3, 128.7, 127.6, 126.5, 126.3, 124.8, 124.5, 123.0, 111.7, 77.9, 61.2, 61.1, 56.4, 33.3. IR νmax: 3446, 1643, 1511, 1457, 1317, 1244, 1122, 909, 809, 778, 732. ESI-HRMS: m/z calculated for C_{23}H_{23}NO₄Se [M+H]^+ 458.0865, found 458.0862.

5-(((2,6-dimethylphenyl)selanyl)methyl)-2-(naphthalen-1-yl)-4,5-dihydrooxazole (3af): 27.6 mg (yield: 70%, 0.1mmol scale), colorless liquid, eluent (6% ethyl acetate in petroleum ether). ¹H NMR (CDCl₃, 400 MHz) δ 9.16 (d, J = 8.8 Hz, 1H), 7.94-7.92 (m, 2H), 7.86 (d, J = 8.0 Hz, 1H), 7.60-7.53 (m, 1H), 7.52-7.48 (m, 1H), 7.46-7.42 (m, 1H), 7.16-7.09 (m, 3H), 4.29 (dd, J = 15.2 Hz, 1H), 3.95 (dd, J = 14.8 Hz, 1H), 3.11 (dd, J = 12.2 Hz, 1H), 2.90 (dd, J = 12.0 Hz, 1H), 2.61 (s, 6H). ¹³C NMR (CDCl₃, 100 MHz) δ 163.7, 143.5, 133.9, 132.1, 131.3, 130.4, 129.3, 128.9, 128.6, 128.0, 127.5, 126.6, 126.3, 124.8, 124.5, 78.4, 61.3, 31.6, 24.8. IR νmax: 3442, 1643, 1511, 1457, 1317, 1244, 1191, 1122, 984, 808, 775. ESI-HRMS: m/z calculated for C_{22}H_{21}NOSe [M+H]^+ 396.0861, found 396.0861.
5-((benzo[d][1,3]dioxol-5-yldiselenyl)methyl)-2-(naphthalen-1-yl)-4,5-dihydrooxazole (3ag): 29.7 mg (yield: 73%, 0.1mmol scale), colorless liquid, eluent (6% ethyl acetate in petroleum ether). $^1$H NMR (CDCl$_3$, 400 MHz) $\delta$ 9.16 (d, $J$ = 8.4 Hz, 1H), 7.99 (d, $J$ = 8.0 Hz, 1H), 7.95 (d, $J$ = 8.0 Hz, 1H), 7.87 (d, $J$ = 8.0 Hz, 1H), 7.60-7.56 (m, 1H), 7.53-7.49 (m, 1H), 7.45 (t, $J$ = 7.8 Hz, 1H), 7.12-7.10 (m, 2H), 6.71 (d, $J$ = 8.0 Hz, 1H), 5.94-5.91 (m, 2H), 4.22-4.85 (m, 1H), 4.31 (dd, $J$ = 14.8 Hz, 1H), 3.98 (dd, $J$ = 14.8 Hz, 1H), 3.26 (dd, $J$ = 12.4 Hz, 1H), 3.05 (dd, $J$ = 12.6 Hz, 1H). $^{13}$C NMR (CDCl$_3$, 100 MHz) $\delta$ 163.8, 148.3, 148.1, 133.9, 132.2, 131.4, 129.3, 128.8, 128.6, 127.5, 126.6, 126.3, 124.8, 124.5, 120.0, 115.2, 109.4, 101.5, 78.0, 61.1, 33.3. IR $\nu_{\text{max}}$: 3444, 1642, 1475, 1318, 1233, 1037, 984, 807, 777. ESI-HRMS: m/z calculated for C$_{21}$H$_{17}$NO$_3$Se [M+H]$^+$ 412.0446, found 412.0443.

2-(naphthalen-1-yl)-5-((naphthalen-1-ylselenyl)methyl)-4,5-dihydrooxazole (3ah): 29.3 mg (yield: 71%, 0.1mmol scale), colorless liquid, eluent (6% ethyl acetate in petroleum ether). $^1$H NMR (CDCl$_3$, 400 MHz) $\delta$ 9.11 (d, $J$ = 8.4 Hz, 1H), 8.48 (d, $J$ = 8.8 Hz, 1H), 7.93-7.89 (m, 3H), 7.85-7.81 (m, 3H), 7.58-7.54 (m, 2H), 7.53-7.47 (m, 2H), 7.43-7.37 (m, 1H), 7.36-7.33 (m, 1H), 4.86-4.82 (m, 1H), 4.27 (dd, $J$ = 15.2 Hz, 1H), 3.98 (dd, $J$ = 15.2 Hz, 1H), 3.36 (dd, $J$ = 12.6 Hz, 1H), 3.13 (dd, $J$ = 12.6 Hz, 1H). $^{13}$C NMR (CDCl$_3$, 100 MHz) $\delta$ 163.7, 134.8, 134.3, 134.2, 133.9, 132.1, 131.3, 129.4, 129.3, 129.0, 128.6, 128.3, 128.0, 127.5, 127.2, 126.6, 126.3, 126.0, 124.8, 124.5, 78.0, 61.2, 32.4. IR $\nu_{\text{max}}$: 3443, 1642, 1511, 1318, 1244, 1191, 1122, 985, 789, 772. ESI-HRMS: m/z calculated for C$_{24}$H$_{19}$NOSe [M+H]$^+$ 418.0705, found 418.0704.
5-((cyclohexylselanyl)methyl)-2-(naphthalen-1-yl)-4,5-dihydrooxazole (3ai): 26.0 mg (yield: 70%, 0.1mmol scale), colorless liquid, eluent (6% ethyl acetate in petroleum ether). $^1$H NMR (CDCl$_3$, 400 MHz) $\delta$ 9.18 (d, $J = 8.8$ Hz, 1H), 8.11 (dd, $J = 7.2$ Hz, 1H), 7.97 (d, $J = 8.4$ Hz, 1H), 7.89 (d, $J = 8.0$ Hz, 1H), 7.63-7.59 (m, 1H), 7.55-7.48 (m, 2H), 5.00-4.92 (m, 1H), 4.36 (dd, $J = 14.8$ Hz, 1H), 4.02 (dd, $J = 15.0$ Hz, 1H), 3.07-3.01 (m, 2H), 2.85 (dd, $J = 12.4$ Hz, 1H), 2.07-2.05 (m, 2H), 1.75-1.63 (m, 2H), 1.55-1.52 (m, 3H), 1.33-1.27 (m, 3H). $^{13}$C NMR (CDCl$_3$, 100 MHz) $\delta$ 163.74, 134.0, 132.1, 131.4, 129.2, 128.6, 127.5, 126.6, 126.3, 124.8, 124.7, 78.7, 61.4, 39.7, 34.9, 27.0, 26.6, 25.9. IR $\nu_{\text{max}}$: 3440, 2928, 2851, 1642, 1590, 1511, 1447, 1317, 1243, 1190, 1122, 1076, 984, 808, 776. ESI-HRMS: m/z calculated for C$_{20}$H$_{23}$NOSe $[M+H]^+$ 374.1018, found 374.1016.

5-((methylselanyl)methyl)-2-(naphthalen-1-yl)-4,5-dihydrooxazole (3aj): 22.8 mg (yield: 75%, 0.1mmol scale), colorless liquid, eluent (6% ethyl acetate in petroleum ether). $^1$H NMR (CDCl$_3$, 400 MHz) $\delta$ 9.16 (d, $J = 8.8$ Hz, 1H), 8.09 (dd, $J = 7.2$ Hz, 1H), 7.96 (d, $J = 8.4$ Hz, 1H), 7.88 (d, $J = 8.4$ Hz, 1H), 7.62-7.57 (m, 1H), 7.54-7.46 (m, 2H), 4.99-4.91 (m, 1H), 4.36 (dd, $J = 15.2$ Hz, 1H), 3.99 (dd, $J = 14.8$ Hz, 1H), 2.97 (dd, $J = 12.8$ Hz, 1H), 2.83 (dd, $J = 12.8$ Hz, 1H), 2.11 (s, 3H). $^{13}$C NMR (CDCl$_3$, 100 MHz) $\delta$ 163.8, 134.0, 132.2, 131.4, 129.2, 128.7, 127.5, 126.6, 126.3, 124.8, 124.7, 78.5, 61.4, 29.7, 5.3. IR $\nu_{\text{max}}$: 3443, 1641, 1590, 1510, 1316, 1243, 1190, 1121, 1075, 983, 807, 775. ESI-HRMS: m/z calculated for C$_{15}$H$_{15}$NOSe $[M+H]^+$ 306.0392, found 306.0392.

2-((phenylselanyl)methyl)-1-tosylpyrrolidine (5aa)$^{[3]}$: 38.8 mg (yield: 98%, 0.1mmol
scale), colorless liquid, eluent (5% ethyl acetate in petroleum ether). $^1$H NMR (CDCl$_3$, 400 MHz) $\delta$ 7.60-7.58 (m, 2H), 7.52 (d, $J = 8.0$ Hz, 2H), 7.35-7.29 (m, 3H), 7.23 (d, $J = 8.0$ Hz, 2H), 3.66-3.59 (m, 2H), 3.50-3.47 (m, 1H), 3.15-3.11 (m, 1H), 2.87-2.81 (m, 1H), 2.39 (s, 3H), 1.84-1.78 (m, 1H), 1.69-1.65 (m, 1H), 1.50-1.47 (m, 1H). $^{13}$C NMR (CDCl$_3$, 100 MHz) $\delta$ 143.6, 134.1, 132.6, 129.8, 129.5, 129.4, 127.7, 127.1, 60.0, 50.1, 33.1, 31.2, 24.0, 21.7. IR $\nu_{\text{max}}$: 2974, 1597, 1479, 1345, 1199, 1159, 1092, 986, 913, 817, 739, 669. **EI-MS**: m/z [M+H]$^+$ 396.2.

4,4-dimethyl-3-phenyl-5-((phenylselanyl)methyl)-4,5-dihydroisoxazole (5ba): 23.0 mg (yield: 67%, 0.1mmol scale), white solid, eluent (5% ethyl acetate in petroleum ether). $^1$H NMR (CDCl$_3$, 400 MHz) $\delta$ 7.99-7.97 (m, 2H), 7.60-7.58 (m, 2H), 7.45-7.38 (m, 3H), 7.31-7.30 (m, 3H), 4.29 (dd, $J = 11.6$ Hz, 1H), 3.61 (dd, $J = 12.8$ Hz, 1H), 3.03 (dd, $J = 12.4$ Hz, 1H), 1.58 (s, 3H), 1.51 (s, 3H). $^{13}$C NMR (CDCl$_3$, 100 MHz) $\delta$ 133.9, 130.2, 129.6, 129.0, 128.6, 128.1, 127.5, 126.3, 82.5, 40.8, 24.3, 23.4, 19.4. IR $\nu_{\text{max}}$: 3445, 1636, 1462, 1379, 1333, 1163, 1021, 768, 741, 692. **ESI-HRMS**: m/z calculated for C$_{18}$H$_{19}$NOSe [M+H]$^+$ 346.0705, found 346.0704.

Ethyl-2-phenyl-5-((phenylselanyl)methyl)-4,5-dihydrofuran-3-carboxylate (5ca): 27.5 mg (yield: 71%, 0.1mmol scale), colorless liquid, eluent (3% ethyl acetate in petroleum ether). $^1$H NMR (CDCl$_3$, 400 MHz) $\delta$ 7.71-7.69 (m, 2H), 7.58-7.55 (m, 2H), 7.39-7.32 (m, 3H), 7.28-7.26 (m, 3H), 4.93-4.86 (m, 1H), 4.13 (q, $J = 7.2$ Hz, 2H), 3.34-3.25 (m, 2H), 3.12 (dd, $J = 12.8$ Hz, 1H), 2.95 (dd, $J = 15.2$ Hz, 1H), 1.20 (t, $J = 7.0$ Hz, 3H). $^{13}$C NMR (CDCl$_3$, 100 MHz) $\delta$ 165.4, 164.7, 133.5, 130.5, 130.1, 129.5, 129.4, 129.3, 127.8, 127.6, 102.2, 80.7, 60.0, 37.4, 32.8, 14.4. IR $\nu_{\text{max}}$: 2979, 1702, 1626, 1478, 1342, 1248, 1086, 758, 738, 692. **EI-MS**: m/z [M+H]$^+$ 389.2.
2-((phenylselanyl)methyl)tetrahydrofuran (5da): 20.3 mg (yield: 84%, 0.1 mmol scale), colorless liquid, eluent (3% ethyl acetate in petroleum ether).  
\[ ^1H \text{NMR (CDCl}_3, 400 MHz) \delta 7.53-7.51 \text{ (m, 2H), 7.27-7.21 \text{ (m, 3H), } 4.10-4.07 \text{ (m, 1H), } 3.93-3.88 \text{ (m, 1H), 3.78-3.73 \text{ (m, 1H), } 3.13 \text{ (dd, } J = 12.2 \text{ Hz, 1H), } 2.99 \text{ (dd, } J = 12.0 \text{ Hz, 1H), } 2.07-2.02 \text{ (m, 1H), } 1.95-1.87 \text{ (m 2H), } 1.69-1.59 \text{ (m, 1H).} \] 
\[ ^13C \text{NMR (CDCl}_3, 100 MHz) \delta 132.7, 130.5, 129.2, 127.0, 78.5, 68.5, 33.2, 31.7, 26.1. \] 
\[ \text{IR } \nu_{\text{max}}: 2972, 2866, 1579, 1478, 1437, 1357, 1183, 1058, 1023, 923, 736, 691. \] 
\[ \text{EI-MS: } m/z [M+H]^+ 265.0. \]

2-((phenylselanyl)methyl)-2,3-dihydrobenzofuran (5ea): 28.4 mg (yield: 98%, 0.1 mmol scale), colorless liquid, eluent (3% ethyl acetate in petroleum ether).  
\[ ^1H \text{NMR (CDCl}_3, 400 MHz) \delta 7.55-7.52 \text{ (m, 2H), 7.25-7.24 \text{ (m, 3H), } 7.12-7.06 \text{ (m, 2H), } 6.82 \text{ (t, } J = 7.4 \text{ Hz, 1H), } 6.74 \text{ (d, } J = 8.0 \text{ Hz, 1H), } 4.95-4.88 \text{ (m, 1H), } 3.36-3.28 \text{ (m, 2H), } 3.10-2.97 \text{ (m, 2H).} \] 
\[ ^13C \text{NMR (CDCl}_3, 100 MHz) \delta 159.4, 133.2, 129.6, 129.4, 128.2, 127.5, 126.4, 125.2, 120.7, 109.7, 82.1, 35.7, 32.9. \] 
\[ \text{IR } \nu_{\text{max}}: 3051, 1597, 1479, 1462, 1437, 1229, 1167, 1022, 962, 872, 738, 691. \] 
\[ \text{EI-MS: } m/z [M+H]^+ 291.2. \]

2-((phenylselanyl)methyl)tetrahydro-2H-pyran (5fa): 21.8 mg (yield: 85%, 0.1 mmol scale), colorless liquid, eluent (3% ethyl acetate in petroleum ether).  
\[ ^1H \text{NMR (CDCl}_3, 400 MHz) \delta 7.52-7.49 \text{ (m, 2H), 7.27-7.21 \text{ (m, 3H), } 4.02-3.98 \text{ (m, 1H), } 3.49-3.39 \text{ (m, 2H), } 3.07 \text{ (dd, } J = 12.2 \text{ Hz, 1H), } 2.93 \text{ (dd, } J = 12.0 \text{ Hz, 1H), } 1.79-1.75 \text{ (m, 2H), } 1.55-1.45 \text{ (m, 3H), } 1.33-1.30 \text{ (m, 1H).} \] 
\[ ^13C \text{NMR (CDCl}_3, 100 MHz) \delta 132.5, 131.0, 129.2, 126.9, 68.9, 33.9, 31.9, 25.9, 23.5. \] 
\[ \text{IR } \nu_{\text{max}}: 3444, 2934, 2845, 1637, 1579, 1478, 1437, 1188, 1087, 1049, 1024, 912, 735, 691. \] 
\[ \text{EI-MS: } m/z [M+Na]^+ 279.1. \]
5-((phenylselanyl)methyl)dihydrofuran-2(3H)-one (5ga): 22.3 mg (yield: 87%, 0.1 mmol scale), colorless liquid, eluent (5% ethyl acetate in petroleum ether). $^1$H NMR (CDCl$_3$, 400 MHz) $\delta$ 7.55-7.53 (m, 2H), 7.30-7.26 (m, 3H), 4.66-4.63 (m, 1H), 3.28 (dd, $J$ = 12.8 Hz, 1H), 3.01 (dd, $J$ = 12.8 Hz, 1H), 2.57-2.50 (m, 2H), 2.48-2.37 (m, 1H), 1.97-1.92 (m, 1H). $^{13}$C NMR (CDCl$_3$, 100 MHz) $\delta$ 176.7, 133.4, 129.5, 129.0, 127.8, 79.5, 32.1, 28.9, 27.8. IR $\nu_{\text{max}}$: 3508, 1761, 1634, 1579, 1478, 1338, 1165, 1013, 981, 911, 738, 691. EI-MS: m/z [M+Na]$^+$ 279.2.

3-methyl-5-((phenylselanyl)methyl)dihydrofuran-2(3H)-one (5ha): 24.6 mg (yield: 91%, 0.1 mmol scale), colorless liquid, eluent (5% ethyl acetate in petroleum ether). $^1$H NMR (CDCl$_3$, 400 MHz) $\delta$ 7.57-7.54 (m, 4H), 7.30-7.29 (m, 6H), 4.68-4.62 (m, 1H), 4.53-4.49 (m, 1H), 3.33 (dd, $J$ = 12.4 Hz, 1H), 3.26 (dd, $J$ = 12.8 Hz, 1H), 3.02 (dd, $J$ = 12.8 Hz, 1H), 2.97 (dd, $J$ = 12.4 Hz, 1H), 2.76-2.72 (m, 1H), 2.69-2.61 (m, 2H), 2.28-2.25 (m, 1H), 2.09-2.03 (m, 1H), 1.59 (d, $J$ = 10.4 Hz, 1H), 1.28 (s, 3H), 1.26 (s, 3H). $^{13}$C NMR (CDCl$_3$, 100 MHz) $\delta$ 179.6, 179.0, 133.4, 133.3, 129.5, 129.4, 129.1, 129.0, 127.8, 127.0, 77.3, 77.1, 37.3, 36.2, 35.1, 34.1, 32.0, 31.9, 16.1, 15.2. IR $\nu_{\text{max}}$: 3458, 2975, 1762, 1637, 1579, 1478, 1344, 1178, 995, 925, 739, 692. EI-MS: m/z [M+H]$^+$ 271.1.

3,3-dimethyl-5-((phenylselanyl)methyl)dihydrofuran-2(3H)-one (5ia): 25.3 mg (yield: 89%, 0.1 mmol scale), colorless liquid, eluent (5% ethyl acetate in petroleum ether). $^1$H NMR (CDCl$_3$, 400 MHz) $\delta$ 7.55-7.53 (m, 2H), 7.29-7.27 (m, 3H), 4.61-4.54 (m, 1H), 3.30 (dd, $J$ = 12.8 Hz, 1H), 3.01 (dd, $J$ = 12.8 Hz, 1H), 2.27 (dd, $J$ = 13.0 Hz, 1H), 1.80 (dd, $J$ = 12.8 Hz, 1H), 1.27 (s, 3H), 1.23 (s, 3H). $^{13}$C NMR (CDCl$_3$, 100 MHz) $\delta$ 181.5, 133.4, 129.5, 129.2, 127.8, 76.0, 43.5, 40.9, 32.3, 25.2, 24.8. IR $\nu_{\text{max}}$: 2971, 1769, 1579, 1478, 1368, 1203, 1133, 1004, 912, 734, 692. EI-MS:
m/z [M+H]^+ 285.2.

3-((phenylselanyl)methyl)isobenzofuran-1(3H)-one (5ja)\(^7\): 18.8 mg (yield: 62%, 0.1mmol scale), colorless liquid, eluent (5% ethyl acetate in petroleum ether). \(^1\)H NMR (CDCl\(_3\), 400 MHz) \(\delta\) 7.91 (d, \(J = 8.0\) Hz, 1H), 7.59-7.47 (m, 5H), 7.29-7.21 (m, 3H), 5.64 (t, \(J = 5.6\) Hz, 1H), 3.46 (dd, \(J = 13.0\) Hz, 1H), 3.33 (dd, \(J = 13.2\) Hz, 1H).

\(^{13}\)C NMR (CDCl\(_3\), 100 MHz) \(\delta\) 170.1, 148.8, 134.1, 133.8, 129.7, 129.5, 129.3, 127.9, 126.8, 125.9, 122.6, 79.3, 32.0. IR \(\nu_{\text{max}}\): 3454, 1762, 1634, 1476, 1287, 1214, 1061, 961, 911, 739, 691. EI-MS: m/z [M+Na]^+ 327.1.

5-phenyl-5-((phenylselanyl)methyl)dihydrofuran-2(3H)-one (5ka)\(^8\): 16.9 mg (yield: 51%, 0.1mmol scale), colorless liquid, eluent (5% ethyl acetate in petroleum ether).

\(^1\)H NMR (CDCl\(_3\), 400 MHz) \(\delta\) 7.48-7.40 (m, 2H), 7.39-7.30 (m, 5H), 7.25-7.22 (m, 3H), 3.48 (dd, \(J = 13.6\) Hz, 13.2 Hz, 2H), 2.77-2.63 (m, 2H), 2.60-2.48 (m, 2H). \(^{13}\)C NMR (CDCl\(_3\), 100 MHz) \(\delta\) 176.1, 142.6, 133.3, 130.4, 129.4, 128.8, 128.3, 127.6, 125.0, 88.4, 41.4, 33.7, 29.3. IR \(\nu_{\text{max}}\): 3058, 1778, 1634, 1478, 1449, 1163, 1022, 930, 739, 702. EI-MS: m/z [M+H]^+ 333.1.

6-((phenylselanyl)methyl)tetrahydro-2H-pyran-2-one (5la)\(^4\): 24.8 mg (yield: 92%, 0.1mmol scale), colorless liquid, eluent (5% ethyl acetate in petroleum ether). \(^1\)H NMR (CDCl\(_3\), 400 MHz) \(\delta\) 7.54-7.51 (m, 2H), 7.29-7.26 (m, 3H), 4.45-4.42 (m, 1H), 3.26 (dd, \(J = 13.0\) Hz, 1H), 3.02 (dd, \(J = 12.8\) Hz, 1H), 2.56-2.52 (m, 1H), 2.48-2.39 (m, 1H), 2.17-2.12 (m, 1H), 1.91-1.88 (m, 1H), 1.83-1.76 (m, 1H), 1.60-1.55 (m, 1H).

\(^{13}\)C NMR (CDCl\(_3\), 100 MHz) \(\delta\) 171.14, 132.98, 129.56, 129.44, 127.56, 79.93, 32.34, 29.52, 27.51, 18.5. IR \(\nu_{\text{max}}\): 3454, 2952, 1732, 1579, 1478, 1438, 1240, 1171, 1072,
5-((phenylseleninyl)methyl)-2-(4-(trifluoromethyl)phenyl)-4,5-dihydrooxazole (6a): 35.6 mg (yield: 89%, 0.1mmol scale), white solid, eluent (10% methanol in dichloromethane). 

$$\text{^{1}H NMR (CDCl}_{3}, \text{400 MHz}} \delta 8.04 (d, J = 8.4 \text{ Hz}, 2H), 7.79-7.72 \text{ (m, 6H)}, 7.69 (d, J = 8.0 \text{ Hz}, 2H), 7.61-7.53 \text{ (m, 8H)}, 5.36-5.34 \text{ (m, 1H)}, 5.06-4.99 \text{ (m, 1H)}, 4.28-4.21 \text{ (m, 2H)}, 3.90 \text{ (dd, } J = 15.2 \text{ Hz, 1H)}, 3.70 \text{ (dd, } J = 15.2 \text{ Hz, 1H)}, 3.28-3.12 \text{ (m, 3H)}, 2.99 \text{ (dd, } J = 12.0 \text{ Hz, 1H}).$$

$$\text{^{13}C NMR (CDCl}_{3}, \text{100 MHz}} \delta 162.3, 162.2, 140.3, 139.5, 133.9 \text{ (d, } J = 33.0 \text{ Hz)}, 133.5 \text{ (d, } J = 21.0 \text{ Hz)}, 131.9 \text{ (d, } J = 134.0 \text{ Hz)}, 131.7 \text{ (d, } J = 131.0 \text{ Hz)}, 130.1, 130.0, 128.8, 128.7, 126.2, 125.8, 125.6 \text{ (q, } J = 4.0 \text{ Hz)}, 125.5 \text{ (q, } J = 4.0 \text{ Hz)}, 125.2, 122.5, 74.1, 73.4, 60.8, 60.6, 58.1, 55.5.$$

**ESI-HRMS:** m/z calculated for C_{25}H_{24}F_{3}NO_{6}Se [M+H]^+ 573.0827, found 573.0834.

N-(2-hydroxy-3-(phenylselenanyl)propyl)-4-(trifluoromethyl)benzamide (6b): 32.6 mg (yield: 81%, 0.1mmol scale), white solid, eluent (30% ethyl acetate in petroleum ether). 

$$\text{^{1}H NMR (d^6DMSO, 400 MHz}} \delta 8.06 \text{ (t, } J = 8.0 \text{ Hz, 1H)}, 7.86 \text{ (d, } J = 8.0 \text{ Hz, 2H)}, 7.49 \text{ (d, } J = 1.6 \text{ Hz, 2H)}, 7.48-7.46 \text{ (m, 2H)}, 7.27-7.18 \text{ (m, 3H)}, 5.36 \text{ (d, } J = 5.2 \text{ Hz, 1H)}, 3.94-3.86 \text{ (m, 1H)}, 3.48-3.44 \text{ (m, 1H)}, 3.42-3.33 \text{ (m, 1H)}, 3.14 \text{ (dd, } J = 12.4 \text{ Hz, 1H)}, 3.03 \text{ (dd, } J = 12.0 \text{ Hz, 1H}).$$

$$\text{^{13}C NMR (d^6DMSO, 100 MHz}} \delta 165.4, 138.3, 131.2 \text{ (d, } J = 19.0 \text{ Hz)}, 130.9, 129.2, 128.2 \text{ (d, } J = 192.0 \text{ Hz)}, 125.3 \text{ (q, } J = 4.0 \text{ Hz)}, 122.6, 68.7, 45.5, 32.5.$$

**ESI-HRMS:** m/z calculated for C_{17}H_{16}F_{3}NO_{2}Se [M+Na]^+ 427.0224, found 427.0233.
5. References

[1] Zhang, X. W.; Cao, B. N.; Yu, S.C.; Zhang, X. M. Angew. Chem. Int. Ed. 2010, 49, 4047.

[2] Tiecco, M.; Testaferri, L.; Tingoli, M.; Bartoli, D.; Balducci, R. J. Org. Chem. 1990, 55, 429.

[3] Ni, Y.; Zuo, H. H.; Li, Y.; Wu, Y. Z.; Zhong, F. G. Org. Lett. 2018, 20, 4350.

[4] Tiecco, M.; Testaferri, L.; Temperini, A.; Bagnoli, L.; Marini, F.; Santi, C. Synlett 2001, 1767.

[5] Shi, H. W.; Yu, C.; Zhu, M.; Yan, J. Synthesis 2016, 48, 57.

[6] Tiecco, M.; Testaferri, L.; Tingoli, M.; Bartol, D. Terrahedron 1989, 45, 6819.

[7] Zhou, E. S.; Han, X.; Yuanyuan Li, Y. Y.; Guo, C.; Dong, C. E. Heterocycles 2015, 91, 1628.

[8] Niu, W. X.; Yeung, Y. –Y. Org. Lett. 2015, 17, 1660.

6. NMR Spectra

6.1 NMR Spectra of Starting Materials
6.2 NMR Spectra of Products

3aa

N
O
SePh

N
O
SePh

3aa
3da

3da
