

**Catalyst-Free Room-Temperature Decarboxylative Tri- or  
Tetrafunctionalization of Alkynyl Carboxylic Acids with N-  
fluorobenzenesulfonimide (NFSI) and Diselenides**

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## Experimental

**General methods and materials.** Proton nuclear magnetic resonance spectra ( $^1\text{H}$  NMR) and carbon nuclear magnetic resonance spectra ( $^{13}\text{C}$  NMR) were recorded at 400 MHz and 100 MHz or 500 MHz and 125 MHz, respectively, using  $\text{CDCl}_3$  as reference standard ( $\delta$  7.26 ppm) for  $^1\text{H}$  NMR and ( $\delta$  77.04 ppm) for  $^{13}\text{C}$  NMR. HRMS (ESI-TOF) were recorded using ESI. Melting points were uncorrected. Precoated silica gel plates F-254 were used for analytical thin-layer chromatography. Column chromatography was performed on silica gel (300-400 mesh). Starting materials alkynyl carboxylic acids and diselenides were readily prepared according to literature procedures. Unless otherwise noted, all reagents were obtained commercially and used without further purification.

**General procedure for the synthesis of polyseleno-substituted enamines 3:** To a stirred solution of alkynyl carboxylic acids (0.5 mmol) and diselenides (0.5 mmol) in dry 1,4-dioxane (2.0 mL) was added NFSI (0.5 mmol) at room temperature for 30 min. The progress of the reaction was monitored by TLC. After the reaction was complete, the reaction mixture was subjected to column chromatographic separation to give pure polyseleno-substituted enamines **3**.

## Spectral data of all compounds

**3a**: White solid, mp. 162-164 °C;  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.93 (dd,  $J = 8.5, 1.1$  Hz, 4H), 7.68–7.63 (m, 2H), 7.59–7.54 (m, 2H), 7.42–7.37 (m, 4H), 7.36–7.22 (m, 10H), 7.21 (d,  $J = 4.3$  Hz, 4H) ppm;  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  139.7, 139.2, 139.1, 137.0, 134.5, 134.2, 133.9, 132.0, 131.9, 131.4, 129.5, 129.2, 128.7, 128.6, 128.5, 128.1, 128.0, 127.8 ppm; HRMS (ESI-TOF) ( $m/z$ ):  $[\text{M} + \text{Na}]^+$  calcd for  $\text{C}_{23}\text{H}_{25}\text{NNaO}_4\text{S}_2\text{Se}_2$ , 685.9453; found 685.9439.

**3b**: Faint yellow oil;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.37 (dd,  $J = 8.6, 1.1$  Hz, 2H),

7.73–7.60 (m, 4H), 7.53–7.49 (m, 2H), 7.45–7.36 (m, 3H), 7.35–7.30 (m, 1H), 7.29–7.18 (m, 7H), 7.16–7.11 (m, 4H), 6.98–6.94 (m, 1H), 2.62 (s, 3H) ppm;  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  140.7, 138.9, 136.8, 134.9, 134.7, 134.2, 133.4, 131.2, 130.4, 130.1, 129.6, 128.9, 128.7, 128.6, 128.5, 128.5, 128.2, 128.1, 127.7, 125.9, 20.7 ppm; **HRMS** (ESI-TOF) (m/z):  $[\text{M} + \text{Na}]^+$  calcd for  $\text{C}_{33}\text{H}_{27}\text{NNaO}_4\text{S}_2\text{Se}_2$ , 747.9610; found 747.9599.

**3c**: White solid, m.p. 152–154 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.89 (d,  $J = 7.9$  Hz, 4H), 7.55–7.49 (m, 4H), 7.36 (t,  $J = 7.8$  Hz, 4H), 7.27 (m, 5H), 7.18 (t,  $J = 6.9$  Hz, 6H), 6.75 (d,  $J = 8.6$  Hz, 2H), 3.80 (s, 3H) ppm;  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  139.8, 139.5, 138.5, 137.8, 136.8, 134.5, 134.3, 133.9, 132.2, 132.1, 131.6, 130.0, 129.5, 128.9, 128.7, 128.5, 128.4, 128.0, 127.9, 127.8, 21.4 ppm; **HRMS** (ESI-TOF) (m/z):  $[\text{M} + \text{Na}]^+$  calcd for  $\text{C}_{33}\text{H}_{27}\text{NNaO}_4\text{S}_2\text{Se}_2$ , 747.9610; found 747.9601.

**3d**: Yellow oil;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.87 (d,  $J = 7.6$  Hz, 4H), 7.50 (t,  $J = 8.1$  Hz, 4H), 7.36–7.27 (m, 6H), 7.27–7.11 (m, 8H), 7.05 (d,  $J = 8.1$  Hz, 2H), 2.62 (q,  $J = 7.6$  Hz, 2H), 1.23 (t,  $J = 7.6$  Hz, 3H) ppm;  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  145.8, 139.8, 139.7, 137.9, 134.5, 134.3, 133.9, 132.2, 131.5, 129.5, 128.7, 128.5, 128.5, 127.9, 127.8, 127.6, 28.8, 15.8 ppm; **HRMS** (ESI-TOF) (m/z):  $[\text{M} + \text{Na}]^+$  calcd for  $\text{C}_{34}\text{H}_{29}\text{NNaO}_4\text{S}_2\text{Se}_2$ , 761.9766; found 761.9760.

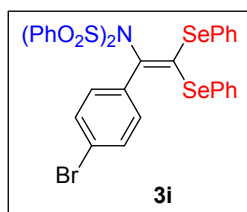
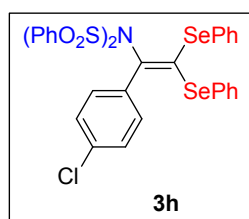
**3e**: Faint yellow solid, m.p. 151–153 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.89 (d,  $J = 7.9$  Hz, 4H), 7.56–7.48 (m, 4H), 7.36 (t,  $J = 7.8$  Hz, 4H), 7.31–7.23 (m, 4H), 7.19 (t,  $J = 6.9$  Hz, 6H), 6.75 (d,  $J = 8.6$  Hz, 2H), 3.80 (s, 3H) ppm;  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  160.3, 139.8, 139.5, 137.1, 134.4, 134.3, 133.8, 133.0, 132.2, 132.1, 129.5, 129.4, 128.6, 128.6, 128.5, 127.9, 127.8, 113.4, 55.4 ppm; **HRMS** (ESI-TOF) (m/z):  $[\text{M} + \text{Na}]^+$  calcd for  $\text{C}_{33}\text{H}_{27}\text{NNaO}_5\text{S}_2\text{Se}_2$ , 763.9559; found 763.9542.

**3f**: Faint yellow solid, m.p. 137–140 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.21–7.96 (m, 3H), 7.73–7.68 (m, 1H), 7.66–7.38 (m, 6H), 7.34–7.10 (m, 12H), 7.05–6.96 (m, 2H) ppm;  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  160.3 (d,  $^1J_{\text{CF}} = 248$  Hz), 144.9, 134.6, 134.0,

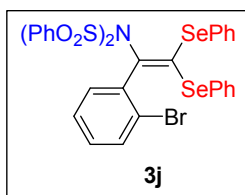
131.4, 131.3, 131.2, 130.9 (d,  $^3J_{CF} = 9.0$  Hz), 130.6, 130.6, 129.5, 128.6, 128.6, 128.5, 128.0, 127.7, 126.3, 126.2, 124.2 (d,  $^4J_{CF} = 3.0$  Hz), 115.0 (d,  $^2J_{CF} = 23.0$  Hz) ppm; **HRMS** (ESI-TOF) (m/z): [M + Na]<sup>+</sup> calcd for C<sub>32</sub>H<sub>24</sub>FNNaO<sub>5</sub>S<sub>2</sub>Se<sub>2</sub>, 751.9359; found 751.9355.

**3g**: Yellow solid, m.p. 155-157 °C; **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>): δ 7.96 (dd,  $J = 8.5, 1.0$  Hz, 4H), 7.66–7.57 (m, 4H), 7.44–7.40 (m, 4H), 7.38–7.17 (m, 10H), 6.97–6.92 (m, 2H) ppm; **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>): δ 163.0 (d,  $^1J_{CF} = 249$  Hz), 139.7, 137.5, 134.6, 134.1, 133.5 (d,  $^3J_{CF} = 9.0$  Hz), 133.3 (d,  $^4J_{CF} = 3.0$  Hz), 131.8, 131.7, 131.5, 129.5, 129.3, 128.8, 128.7, 128.6, 128.2, 127.9, 115.0 (d,  $^2J_{CF} = 22.0$  Hz) ppm; **HRMS** (ESI-TOF) (m/z): [M + Na]<sup>+</sup> calcd for C<sub>32</sub>H<sub>24</sub>FNNaO<sub>5</sub>S<sub>2</sub>Se<sub>2</sub>, 751.9359; found 751.9349.

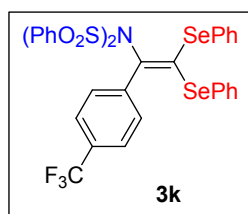
**3h**: Yellow oil; **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>): δ 7.91 (dd,  $J = 8.5, 1.1$  Hz, 4H), 7.57–7.50 (m, 3H), 7.41-7.37 (m, 4H), 7.29 (dd,  $J = 5.0, 1.3$  Hz, 1H), 7.28–7.12 (m, 11H), 7.10 (dd,  $J = 5.0, 3.0$  Hz, 1H) ppm; **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>): δ 139.8, 139.2, 138.0, 134.5, 133.9, 133.8, 133.2, 131.7, 131.6, 130.1, 129.3, 129.2, 128.7, 128.6, 128.5, 128.0, 127.8, 124.6 ppm; **HRMS** (ESI-TOF) (m/z): [M + Na]<sup>+</sup> calcd for C<sub>32</sub>H<sub>24</sub>ClNNaO<sub>5</sub>S<sub>2</sub>Se<sub>2</sub>, 767.9063; found 767.9059.



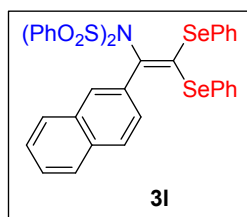
(m, 1H), 7.55–7.47 (m, 1H), 7.55–7.47 (m, 1H), 7.55–7.47 (m, 1H) ppm; **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>): δ 139.7, 136.9, 134.6, 134.5, 134.2, 133.9, 133.8, 131.3, 131.1, 129.4, 129.1, 128.7, 128.6, 128.5, 128.4, 128.0, 127.9, 127.7 ppm; **HRMS** (ESI-TOF) (m/z): [M + Na]<sup>+</sup> calcd for C<sub>36</sub>H<sub>27</sub>BrNNaO<sub>4</sub>S<sub>2</sub>Se<sub>2</sub>, 811.8558, 813.8538; found 811.8541, 813.8533.



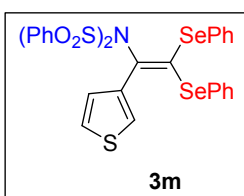
**3j:** Yellow solid, m.p. 161-164 °C;  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.52–7.48 (m, 2H), 7.41–7.39 (m, 2H), 7.38–7.06 (m, 14H), 7.06–6.94 (m, 4H), 6.93–6.85 (m, 2H) ppm;  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  148.7, 140.8, 137.0, 134.0, 132.6, 132.2, 131.8, 131.5, 130.8, 129.5, 128.9, 128.8, 128.7, 128.5, 128.3, 127.6, 127.4, 126.6, 123.1, 116.8 ppm; **HRMS** (ESI-TOF) (m/z):  $[\text{M} + \text{Na}]^+$  calcd for  $\text{C}_{36}\text{H}_{27}\text{BrNNaO}_4\text{S}_2\text{Se}_2$ , 811.8558, 813.8538; found 811.8546, 813.8532.



**3k:** Yellow oil;  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.91 (d,  $J = 7.8$  Hz, 3H), 7.81–7.76 (m, 1H), 7.66 (d,  $J = 8.2$  Hz, 1H), 7.57–7.51 (m, 2H), 7.43–7.28 (m, 9H), 7.27–7.15 (m, 3H), 7.15–7.07 (m, 3H), 7.07–6.96 (m, 2H) ppm;  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  141.8, 140.7, 139.6, 139.2, 136.2, 131.6, 131.5, 131.1, 129.5, 129.4, 129.2, 128.8, 128.7, 128.7, 128.6, 128.4, 127.2, 126.5 (q,  $J_{\text{CF}} = 271.0$  Hz), 124.8 (q,  $J = 4.0$  Hz) ppm; **HRMS** (ESI-TOF) (m/z):  $[\text{M} + \text{Na}]^+$  calcd for  $\text{C}_{33}\text{H}_{24}\text{F}_3\text{NNaO}_4\text{S}_2\text{Se}_2$ , 801.9327; found 801.9320.

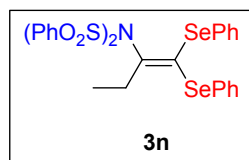


**3l:** Faint yellow solid, m.p. 169-172 °C;  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.95 (s, 1H), 7.88 (d,  $J = 7.9$  Hz, 4H), 7.75 (d,  $J = 8.0$  Hz, 1H), 7.72–7.66 (m, 2H), 7.63 (d,  $J = 8.6$  Hz, 1H), 7.44 (m, 5H), 7.33 (d,  $J = 7.5$  Hz, 2H), 7.30–7.25 (m, 1H), 7.20 (m, 6H), 7.12 (m, 4H) ppm;  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  139.7, 139.2, 138.8, 135.9, 134.6, 134.3, 134.2, 133.8, 133.2, 132.6, 131.9, 131.6, 131.5, 130.4, 129.5, 129.3, 129.2, 128.7, 128.7, 128.5, 128.4, 128.1, 128.0, 127.8, 127.7, 127.5, 127.1, 126.3 ppm; **HRMS** (ESI-TOF) (m/z):  $[\text{M} + \text{Na}]^+$  calcd for  $\text{C}_{36}\text{H}_{27}\text{NNaO}_4\text{S}_2\text{Se}_2$ , 783.9610; found 783.9597.

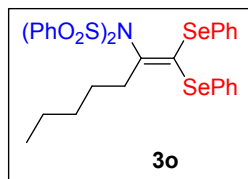


**3m:** Red solid, m.p. 173-176 °C;  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.54–7.50 (m, 3H), 7.38 (dd,  $J = 8.1, 1.2$  Hz, 2H), 7.30–7.21 (m,

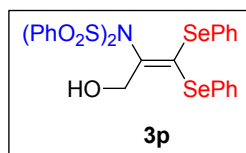
15H), 7.15 (dd,  $J = 7.8, 6.9$  Hz, 2H), 7.06 (t,  $J = 7.6$  Hz, 2H) ppm;  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  144.9, 142.3, 138.0, 136.2, 133.7, 132.8, 132.7, 130.4, 129.3, 129.2, 129.0, 128.7, 128.4, 127.9, 127.6, 127.4, 127.0, 119.6 ppm; HRMS (ESI-TOF) ( $m/z$ ):  $[\text{M} + \text{Na}]^+$  calcd for  $\text{C}_{30}\text{H}_{23}\text{NNaO}_4\text{S}_3\text{Se}_2$ , 739.9017; found 739.9012.



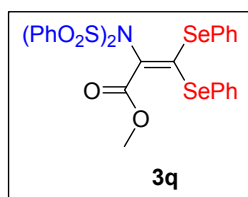
**3n:** White acicular crystal, m.p. 150-152 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.17–7.97 (m, 4H), 7.67–7.59(m, 4H), 7.52–7.42 (m, 6H), 7.37–7.35 (m, 3H), 7.31–7.24 (m, 3H), 1.59 (q,  $J = 7.3$  Hz, 2H), 0.85 (t,  $J = 7.3$  Hz, 3H) ppm;  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  167.3, 139.0, 136.4, 134.1, 131.7, 129.9, 129.6, 129.2, 129.1, 128.9, 128.6, 126.7, 116.8, 26.9, 14.0 ppm; HRMS (ESI-TOF) ( $m/z$ ):  $[\text{M} + \text{Na}]^+$  calcd for  $\text{C}_{23}\text{H}_{25}\text{NNaO}_4\text{S}_2\text{Se}_2$ , 685.9453; found 685.9439.



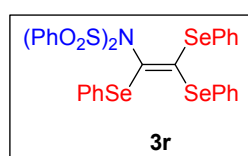
**3o:** White solid, m.p. 157-158 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.10–7.99 (m, 4H), 7.68–7.62 (m, 2H), 7.58 (t,  $J = 7.5$  Hz, 2H), 7.45 (dd,  $J = 8.7, 7.1$  Hz, 6H), 7.36–7.30 (m, 3H), 7.29–7.21 (m, 3H), 1.51 (dd,  $J = 11.3, 4.9$  Hz, 2H), 1.42–1.31 (m, 2H), 0.94–0.82 (m, 2H), 0.74 (dd,  $J = 14.7, 7.4$  Hz, 2H), 0.66 (t,  $J = 7.3$  Hz, 3H) ppm;  $^{13}\text{C}$  NMR (100MHz,  $\text{CDCl}_3$ ):  $\delta$  166.6, 139.0, 136.5, 134.2, 131.6, 130.0, 129.6, 129.1, 129.0, 128.9, 128.6, 126.8, 116.2, 33.6, 31.4, 28.7, 21.7, 13.9 ppm; HRMS (ESI-TOF) ( $m/z$ ):  $[\text{M} + \text{Na}]^+$  calcd for  $\text{C}_{31}\text{H}_{31}\text{NNaO}_4\text{S}_2\text{Se}_2$ , 727.9923; found 727.9912.



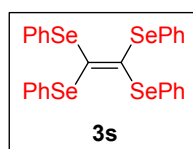
**3p:** Yellow solid, m.p. 155-158 °C;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.03–7.97 (m, 4H), 7.59 (m, 6H), 7.45 (t,  $J = 7.9$  Hz, 4H), 7.39–7.27 (m, 6H), 3.25 (s, 2H), 2.46 (s, 1H) ppm;  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  163.2, 138.0, 136.9, 134.5, 130.7, 130.1, 129.7, 129.5, 129.2, 129.1, 129.0, 127.6, 127.1, 118.7, 59.5 ppm; HRMS (ESI-TOF) ( $m/z$ ):  $[\text{M} + \text{Na}]^+$  calcd for  $\text{C}_{27}\text{H}_{23}\text{NaNO}_4\text{S}_2\text{Se}_2$ , 687.9246; found 687.9233.



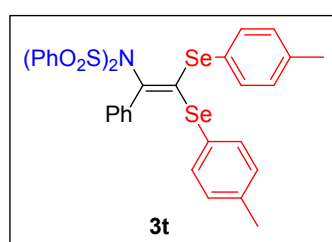
**3q:** Yellow oil;  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.08–8.03 (m, 4H), 7.55–7.47 (m, 6H), 7.43–7.34 (m, 4H), 7.30–7.21 (m, 6H), 2.93 (s, 3H) ppm;  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ )  $\delta$  162.2, 139.1, 135.7, 134.2, 132.8, 130.8, 129.6, 129.2, 129.2, 129.1, 129.0, 128.9, 128.8, 128.7, 127.4, 52.1 ppm; **HRMS** (ESI-TOF) ( $m/z$ ):  $[\text{M} + \text{Na}]^+$  calcd for  $\text{C}_{28}\text{H}_{23}\text{NaNO}_6\text{S}_2\text{Se}_2$ , 715.9195; found 715.9166.



**3r:** Faint yellow acicular crystal, m.p. 157-159 °C;  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.20 (dt,  $J=8.7, 1.7$  Hz, 4H), 7.84–7.77 (m, 2H), 7.69–7.66 (m, 2H), 7.57–7.49 (m, 4H), 7.43–7.36 (m, 2H), 7.35–7.24 (m, 2H), 7.24–7.18 (m, 1H), 7.16–6.99 (m, 8H) ppm;  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  152.7, 139.2, 135.4, 134.4, 133.4, 132.3, 132.1, 131.5, 130.7, 129.6, 129.7, 129.0, 128.4, 128.3, 128.2, 127.6, 127.2, 124.4 ppm; **HRMS** (ESI-TOF) ( $m/z$ ):  $[\text{M} + \text{Na}]^+$  calcd for  $\text{C}_{32}\text{H}_{25}\text{NNaO}_4\text{S}_2\text{Se}_3$ , 733.9453; found 733.9446.

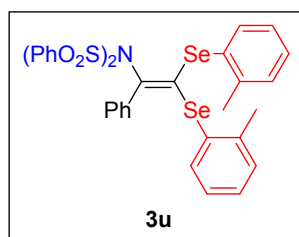


**3s:** Faint yellow solid, m.p. 113-115 °C;  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.27 (m, 12H), 7.19 (t,  $J = 7.2$  Hz, 8H) ppm;  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  133.6, 132.4, 130.8, 128.8, 127.7 ppm; **HRMS** ( $m/z$ ) (APCI): calcd for  $\text{C}_{26}\text{H}_{21}\text{Se}_4$ , 650.8312  $[\text{M}+\text{H}^+]$ ; found 650.8319.

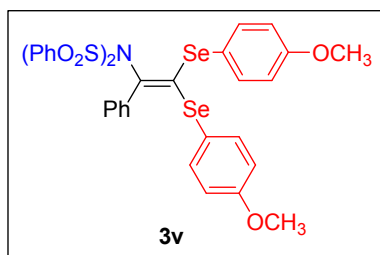


**3t:** Yellow solid, m.p. 108-111 °C;  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.86 (dd,  $J = 8.5, 1.1$  Hz, 4H), 7.62–7.54 (m, 2H), 7.54–7.45 (m, 2H), 7.39–7.15 (m, 9H), 7.07–6.96 (m, 6H), 2.31 (s, 3H), 2.30 (s, 3H);  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  139.7, 138.8, 138.1, 137.7, 137.1, 134.7, 134.2, 133.8, 131.4, 129.4, 129.2, 129.1, 128.6, 128.5, 128.4, 127.9, 21.31, 21.26 ppm; **HRMS** (ESI-TOF) ( $m/z$ ):  $[\text{M} + \text{Na}]^+$  calcd for  $\text{C}_{34}\text{H}_{29}\text{NNaO}_4\text{S}_2\text{Se}_2$ , 761.9766; found

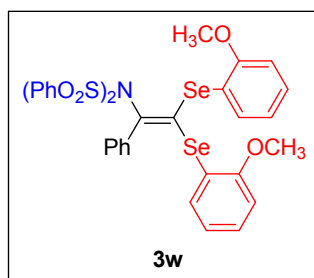
761.9763.



**3u:** Yellow solid, m.p. 124-127 °C;  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.93 (dd,  $J = 8.5, 1.1$  Hz, 4H), 7.69–7.64 (m, 2H), 7.52–7.47 (m, 2H), 7.37–7.23 (m, 8H), 7.13 (qd,  $J = 7.4, 1.3$  Hz, 2H), 7.07–6.91 (m, 5H), 1.93 (s, 3H), 1.74 (s, 3H) ppm;  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  140.9, 140.5, 140.4, 139.8, 137.7, 137.3, 135.6, 135.4, 133.7, 132.6, 132.3, 131.0, 129.5, 129.4, 129.3, 129.1, 128.4, 128.3, 128.1, 127.9, 126.1, 125.8, 22.1, 21.8 ppm; **HRMS** (ESI-TOF) ( $m/z$ ):  $[\text{M} + \text{Na}]^+$  calcd for  $\text{C}_{34}\text{H}_{29}\text{NNaO}_4\text{S}_2\text{Se}_2$ , 761.9766; found 761.9756.



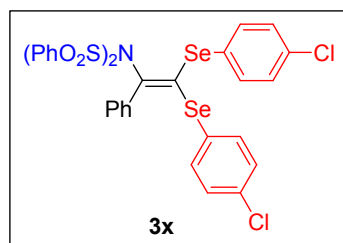
**3v:** Yellow oil;  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.86 (dd,  $J = 8.5, 1.1$  Hz, 3H), 7.57–7.51 (m, 4H), 7.38–7.33 (m, 4H), 7.32–7.19 (m, 6H), 7.11–7.05 (m, 2H), 6.79–6.68 (m, 4H), 3.80 (s, 6H) ppm;  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  159.9, 159.6, 140.8, 139.8, 138.1, 137.1, 136.4, 136.1, 133.8, 131.4, 129.5, 129.1, 128.5, 128.1, 122.7, 122.5, 114.3, 114.1, 55.3, 55.3 ppm; **HRMS** (ESI-TOF) ( $m/z$ ):  $[\text{M} + \text{Na}]^+$  calcd for  $\text{C}_{34}\text{H}_{29}\text{NNaO}_6\text{S}_2\text{Se}_2$ , 793.9664; found 793.9659.



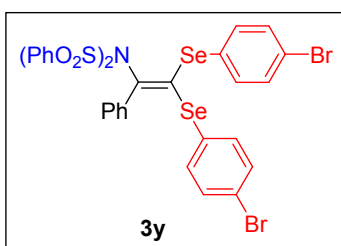
**3w:** Faint yellow solid, m.p. 104-107 °C;  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.94 (dd,  $J = 8.5, 1.1$  Hz, 4H), 7.67–7.62 (m, 2H), 7.47–7.42 (m, 2H), 7.31–7.25 (m, 5H), 7.22–7.09 (m, 6H), 6.82–6.78 (m, 1H), 6.74–6.65 (m, 2H), 6.60 (dd,  $J = 8.2, 0.8$  Hz, 1H), 3.65 (s, 3H), 3.64 (s, 3H) ppm;  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  158.0, 157.8, 140.0, 138.9, 137.6, 137.0, 134.8, 134.5, 133.5, 130.9, 129.6, 128.8, 128.8, 128.5, 128.1, 127.8, 121.3, 120.9,



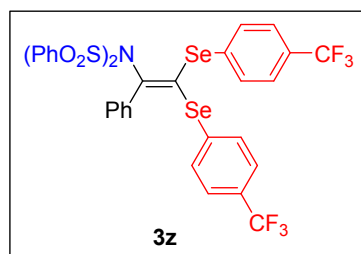
120.8, 120.7, 110.2, 110.1, 55.50, 55.45 ppm; **HRMS** (ESI-TOF) ( $m/z$ ):  $[M + Na]^+$  calcd for  $C_{34}H_{29}NNaO_6S_2Se_2$ , 793.9664; found 793.9656.



**3x:** Yellow solid, m.p. 110-113 °C;  **$^1H$  NMR** (400 MHz,  $CDCl_3$ ):  $\delta$  7.86 (d,  $J = 7.9$  Hz, 4H), 7.55 (dd,  $J = 11.8, 7.4$  Hz, 4H), 7.40–7.29 (m, 5H), 7.28–7.21 (m, 4H), 7.19–7.14 (m, 4H), 7.07 (d,  $J = 8.4$  Hz, 2H) ppm;  **$^{13}C$  NMR** (100 MHz,  $CDCl_3$ ):  $\delta$  139.9, 139.5, 137.9, 136.6, 135.7, 135.5, 134.5, 134.3, 134.0, 131.2, 129.9, 129.9, 129.5, 129.4, 128.9, 128.7, 128.6, 128.2 ppm; **HRMS** (ESI-TOF) ( $m/z$ ):  $[M + Na]^+$  calcd for  $C_{32}H_{23}Cl_2NNaO_4S_2Se_2$ , 801.8674; found 801.8656.

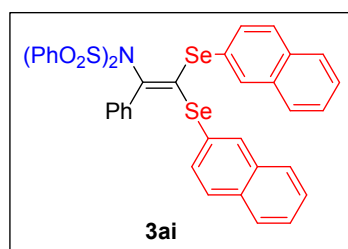


**3y:** Faint yellow solid, m.p. 128-130 °C;  **$^1H$  NMR** (400 MHz,  $CDCl_3$ ):  $\delta$  7.86 (dd,  $J = 8.5, 1.1$  Hz, 3H), 7.59–7.53 (m, 4H), 7.40–7.29 (m, 9H), 7.29–7.22 (m, 4H), 7.19–7.14 (m, 2H), 7.06–6.98 (m, 2H) ppm;  **$^{13}C$  NMR** (100 MHz,  $CDCl_3$ ):  $\delta$  140.0, 139.6, 137.6, 136.6, 135.9, 135.7, 134.0, 131.9, 131.7, 131.3, 130.7, 130.6, 129.5, 129.4, 128.6, 128.2, 122.7, 122.5 ppm; **HRMS** (ESI-TOF) ( $m/z$ ):  $[M + Na]^+$  calcd for  $C_{32}H_{23}Br_2NNaO_4S_2Se_2$ , 889.7651, 891.7643; found 889.7656, 891.7639.



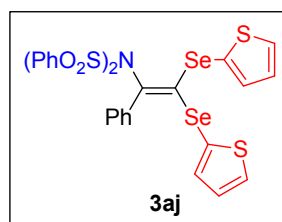
**3z:** Yellow solid, m.p. 117-119 °C;  **$^1H$  NMR** (400 MHz,  $CDCl_3$ ):  $\delta$  7.91–7.85 (m, 4H), 7.60–7.54 (m, 4H), 7.47–7.30 (m, 11H), 7.26–7.23 (m, 4H) ppm;  **$^{13}C$  NMR** (100 MHz,  $CDCl_3$ ):  $\delta$  141.1, 139.5, 136.5, 136.4, 136.3, 135.9, 134.1, 134.0, 133.9, 131.2, 130.7, 130.3, 129.9, 129.4, 128.6, 128.3, 125.5 (q,  $J = 4.0$  Hz), 125.3 (q,  $J = 4.0$  Hz) ppm; **HRMS** (ESI-TOF) ( $m/z$ ):  $[M + Na]^+$  calcd for  $C_{34}H_{23}F_6NNaO_4S_2Se_2$ , 869.9201; found

869.9194.



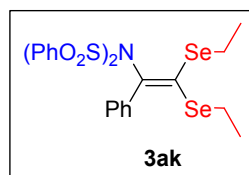
**3ai:** Gray solid, m.p. 108-110 °C; **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>): δ 7.84 (dd, *J* = 8.5, 1.1 Hz, 4H), 7.80–7.75 (m, 3H), 7.71–7.68 (m, 3H), 7.59–7.55 (m, 2H), 7.48–7.37 (m, 5H), 7.37–7.16 (m, 10H), 6.93–6.74 (m, 2H); **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>): δ 140.3, 139.8, 138.9, 137.1,

134.4, 134.2, 134.1, 134.0, 133.8, 133.6, 133.5, 131.3, 131.2, 130.5, 129.5, 129.4, 129.3, 128.8, 128.4, 128.2, 128.1, 128.0, 127.7, 126.9, 126.6, 126.1, 125.9, 125.8, 125.5, 125.0 ppm; **HRMS** (ESI-TOF) (*m/z*): [M + Na]<sup>+</sup> calcd for C<sub>40</sub>H<sub>29</sub>NNaO<sub>4</sub>S<sub>2</sub>Se<sub>2</sub>, 833.9766; found 833.9760.



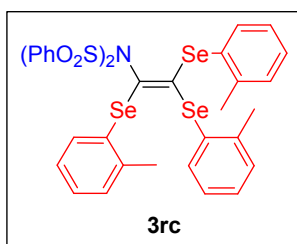
**3aj:** Brown oil; **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>): δ 7.83–7.74 (m, 4H), 7.56–7.50 (m, 4H), 7.49–7.43 (m, 2H), 7.38–7.31 (m, 5H), 7.30–7.22 (m, 4H), 7.01–6.98 (m, 1H), 6.93 (d, *J* = 2.3 Hz, 1H) ppm; **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>): δ 140.4, 139.6,

137.0, 136.6, 136.5, 133.9, 131.8, 131.7, 131.6, 129.5, 129.3, 129.3, 128.7, 128.2, 127.7, 127.4, 126.1, 125.9. **HRMS** (ESI-TOF) (*m/z*): [M + Na]<sup>+</sup> calcd for C<sub>23</sub>H<sub>21</sub>NNaO<sub>4</sub>S<sub>4</sub>Se<sub>2</sub>, 745.8582; found 745.8574.

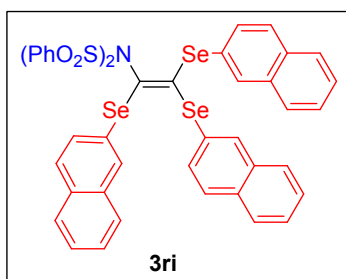


**3ak:** Faint yellow solid, m.p. 108-111 °C; **<sup>1</sup>H NMR** (400 MHz, CDCl<sub>3</sub>): δ 7.84 (d, *J* = 7.9 Hz, 4H), 7.62–7.59 (m, 2H), 7.55 (t, *J* = 7.4 Hz, 2H), 7.38 (t, *J* = 7.8 Hz, 4H), 7.30–7.23 (m, 3H), 2.76 (q, *J* = 7.5 Hz, 2H), 2.61 (q, *J* = 7.4 Hz, 2H), 1.35–1.31 (m,

6H) ppm; **<sup>13</sup>C NMR** (100 MHz, CDCl<sub>3</sub>): δ 140.2, 137.9, 136.9, 133.6, 131.3, 129.4, 128.9, 128.8, 128.4, 128.1, 25.9, 25.6, 15.4, 14.9 ppm; **HRMS** (ESI-TOF) (*m/z*): [M + Na]<sup>+</sup> calcd for C<sub>24</sub>H<sub>25</sub>NNaO<sub>4</sub>S<sub>2</sub>Se<sub>2</sub>, 637.9453; found 637.9441.



**3rc:** Yellow oil;  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  8.15 (t,  $J=8.2$  Hz, 5H), 7.93 (d,  $J=7.6$  Hz, 1H), 7.61 (t,  $J=7.6$  Hz, 2H), 7.53–7.41 (m, 6H), 7.23–7.05 (m, 6H), 6.87–6.78 (m, 2H), 2.32 (s, 3H), 1.95 (s, 3H), 1.58 (s, 3H) ppm;  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  141.8, 139.3, 137.2, 136.7, 134.4, 134.2, 134.0, 133.5, 132.8, 132.1, 130.9, 130.6, 129.6, 129.5, 129.2, 129.1, 128.9, 128.8, 128.1, 127.5, 127.2, 126.8, 126.0, 125.6, 22.4, 21.8, 21.4 ppm; **HRMS** (ESI-TOF) (m/z):  $[\text{M} + \text{Na}]^+$  calcd for  $\text{C}_{35}\text{H}_{31}\text{NNaO}_4\text{S}_2\text{Se}_3$ , 853.9096; found 853.9099.



**3ri:** White solid, m.p. 96-98 °C;  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  7.89–7.79 (m, 13H), 7.62 (ddd,  $J = 8.4, 6.9, 1.3$  Hz, 2H), 7.59–7.52 (m, 6H), 7.46–7.34 (m, 10H) ppm;  $^{13}\text{C NMR}$  (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  139.3, 134.4, 134.2, 133.9, 133.9, 132.8, 130.2, 129.1, 128.9, 128.1, 127.6, 127.4, 126.7, 126.2, 126.1, 120.8 ppm; **HRMS** (ESI-TOF) (m/z):  $[\text{M} + \text{Na}]^+$  calcd for  $\text{C}_{44}\text{H}_{31}\text{NNaO}_4\text{S}_2\text{Se}_3$ , 961.9096; found 961.9097.

**Copies of  $^1\text{H NMR}$  and  $^{13}\text{C NMR}$  Spectra of all compounds**

