# **Supporting Information**

# I. General method

All chemicals and solvents were purified according to the standard procedure.<sup>[1]</sup> The trichalcogenasumanenes (**3S** and **3Se**) were synthesized according to our previous report,<sup>[2]</sup> and the triazacoronene was synthesized according to the method reported by Wei *et al*.<sup>[3]</sup>

Melting points were determined on WRS-2 melting point apparatus. <sup>1</sup>H, and <sup>13</sup>C NMR spectra were recorded on a Bruker Avance III 400MHz (100 MHz for <sup>13</sup>C) or a VARIAN INOVA 600MHz spectrometer (125 MHz for <sup>13</sup>C) spectrometer. The chemical shifts of <sup>1</sup>H and <sup>13</sup>C NMR were recorded using TMS as internal standard. High-resolution mass spectral analysis (HRMS) was carried out on a maXis 4G mass spectrometer (Bruker Daltonic Inc.). The UV-vis spectra in CH<sub>2</sub>Cl<sub>2</sub> solution ( $1.0 \times 10^{-5}$  mol L<sup>-1</sup>) were measured at 20 °C on the UV-2600 UV-Vis spectrometer (Shimadzu). The infrared (IR) spectra were record on the PerkinElmer Spectrum 400 spectrometer with the resolution of 2 cm<sup>-1</sup>. Fluorescence excitation and emission spectra were recorded with an RF-5301(pc)s Spectrofluorophotometer, fluorescence lifetime and steady state were measured on FLS920 Spectrofluorophotometer. All calculations were carried out with the *Gaussian 09* suite of programs.<sup>[S4]</sup> For DFT calculations, we used the hybrid gradient corrected exchange functional of Lee, Yang, and Parr. A standardized 6-311G basis set was used together with polarization (d) and (p) functions.<sup>[S5]</sup> The crystal structure was solved by a direct method *SIR2004*<sup>[S6]</sup> and refined by full-matrix least-square method on *F*<sup>2</sup> by means of *SHELXL-97*<sup>[S7]</sup>. The calculated positions of the hydrogen atoms were included in the final refinement.

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# **II. Synthesis**



Scheme S1. The chemical structures of the compounds in this report.

### 2.1 Synthesis of 1a from HBT



To a hexane (40 mL) solution of 2,3,6,7,10,11-hexabutoxytriphenylene (**HBT**) (6.6 g, 0.01 mol) was added TMEDA (6 mL, 0.04 mol) and *n*-butyllithium (2.4 M in hexane,16.6 mL, 0.04mol) at 60  $^{\circ}$ C for 3 hours. The resulting solution was cooled to -78  $^{\circ}$ C and diluted with 60 mL THF. Sulfur powder (2.56 g, 0.08 mol) was added in one portion. The resulting orange mixture was slowly warmed to room temperature and allowed to stir overnight. The reaction was quenched by adding distilled water and then extracted with CH<sub>2</sub>Cl<sub>2</sub> (3×50 mL). The organic layers were combined and dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, then concentrated under reduced pressure. The crude product was further purified by column chromatography on silica (eluent, CH<sub>2</sub>Cl<sub>2</sub>: petro ether, 1:3,  $\nu/\nu$ ) to afford **1a** as an orange-yellow solid (6.8 g, yield 90 %). For **1a**, mp: 107-108  $^{\circ}$ C; <sup>1</sup>H NMR (400 MHz, Chloroform-*d*):  $\delta$ (ppm) 7.78 (s, 1H), 7.77 (s, 1H), 4.41-4.35 (m, 4H), 4.31-4.20 (m, 8H), 2.00-1.82 (m, 12H), 1.70-1.56 (m, 12H), 1.10-1.00 (m, 18H); <sup>13</sup>C NMR (100 MHz, Chloroform-*d*):  $\delta$ (ppm) 152.61, 152.14, 146.30, 146.07, 144.79, 143.05, 130.46, 128.04, 126.90, 126.22, 123.61, 123.21, 122.30, 121.07, 120.27, 105.60, 104.80, 74.54, 73.64, 73.08, 73.01, 70.08, 68.63, 32.51, 32.49, 32.46, 19.45, 19.38, 19.33, 19.29, 14.00, 13.98, 13.96, 13.93, 13.92; HRMS: calculated for C4<sub>2</sub>H<sub>56</sub>O<sub>6</sub>S<sub>3</sub> +H<sup>+</sup>, 753.3312; found, 753.3279.

### 2.2 Synthesis of 1b from HBT



To a hexane (40 mL) solution of **HBT** (6.6 g, 0.01 mol) was added TMEDA (6 mL, 0.04 mol) and *n*-butyllithium (2.4 M in hexane, 16.6 mL, 0.04mol) at 60 °C for 3 hours. The resulting solution was

cooled to -78 °C and diluted with 60 mL THF. Selenium powder (6.32g, 0.08 mol) was added in one portion. The resulting yellow solution was slowly warmed to room temperature and stirred overnight. The reaction was quenched by adding distilled water and extracted with CH<sub>2</sub>Cl<sub>2</sub> (3×50 mL). The organic layers were combined and dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, then concentrated under reduced pressure. The crude product was further purified by column chromatography on silica (eluent, CH<sub>2</sub>Cl<sub>2</sub>: petro ether, 1:3,  $\nu/\nu$ ) to afford **1b** as an orange-yellow solid (7.15 g, yield 80%) and **2b** (yellow powder, 0.46 g, 5.6 %). For **1b**, mp: 113.0-114.7 °C; <sup>1</sup>H NMR (400 MHz, Chloroform-*d*):  $\delta$ (ppm) 7.83 (s, 1H), 7.81 (s, 1H), 4.36-4.25 (m, 12H), 1.98-1.85 (m, 12H), 1.68-1.60 (m, 12H), 1.10-1.03 (m, 18H); <sup>13</sup>C NMR (100 MHz, Chloroform-*d*):  $\delta$ (ppm) 152.67, 152.20, 146.32, 146.13, 144.87, 128.18, 123.28, 122.42, 120.39, 105.71, 104.96, 74.59, 73.71, 73.14, 73.06, 70.19, 68.70, 32.47, 32.45, 32.42, 31.70, 31.42, 19.44, 19.36, 19.31, 19.26, 13.99, 13.97, 13.94, 13.92, 13.90; HRMS: calculated for C<sub>42</sub>H<sub>56</sub>O<sub>6</sub>Se<sub>3</sub>+H<sup>+</sup>, 895.1653; found, 895.1647.

### 2.3 Synthesis of 2a from 1a



The mixture of **1a** (6.5 g, 8.6 mmol) and copper nanopowder (80-100 nm mesh, 2.76 g, 0.043 mol) was slowly heated to 200 °C and kept at this temperature for 2 h under argon. After cooled down to room temperature, the reaction mixture was washed with CH<sub>2</sub>Cl<sub>2</sub>. The solution was concentrated and the crude product was separated by column chromatography on silica (eluent, CH<sub>2</sub>Cl<sub>2</sub>: Petro ether, 1:3, v/v) to give **2a** (pale yellow powder, 4.85 g, 78 %). For **2a**, mp: 132.4–134.2 °C; <sup>1</sup>H NMR (400 MHz, Chloroform-*d*):  $\delta$ (ppm) 7.86 (s, 2H), 4.49-4.38 (m, 8H), 4.29 (t, *J* = 6.4 Hz, 4H), 2.00-1.84 (m, 12H), 1.71-1.59 (m, 12H), 1.11-1.01 (m, 18H); <sup>13</sup>C NMR (100 MHz, Chloroform-*d*):  $\delta$ (ppm) 148.88, 147.34, 146.79, 128.87, 126.29, 124.17, 122.36, 74.81, 74.52, 73.41, 32.56, 32.51, 29.85, 19.49, 19.46, 19.38, 14.13, 14.12, 14.06, 14.05; HRMS: calculated for C<sub>42</sub>H<sub>56</sub>O<sub>6</sub>S<sub>2</sub>+ H<sup>+</sup>, 721.3591; found, 721.3575.

### 2.4 Synthesis of 2b from 1b



The mixture of **1b** (13.76 g, 15.4 mmol) and copper nanopowder (80-100 nm mesh, 4.9 g, 0.077 mol) was heated to 200 °C and kept at this temperature for 2 h under argon. After cooled down to room temperature, the reaction mixture was washed with CH<sub>2</sub>Cl<sub>2</sub>. The solution was concentrated and further purified by column chromatography on silica (eluent, CH<sub>2</sub>Cl<sub>2</sub>) to give **2b** (pale yellow powder, 12.58 g, 100%). For **2b**, mp: 106.5-107.9 °C; <sup>1</sup>H NMR (400 MHz, Chloroform-*d*):  $\delta$ (ppm) 7.88 (s, 2H), 4.40-4.33 (m, 8H), 4.30 (t, *J* = 6.4 Hz, 4H), 2.01-1.83 (m, 12H), 1.70-1.58 (m, 12H), 1.10-1.01 (m, 18H); <sup>13</sup>C NMR (101 MHz, Chloroform-*d*):  $\delta$ (ppm) 151.66, 147.70, 144.98, 131.73, 128.83, 124.93, 122.76, 105.20, 72.86, 72.63, 69.55, 32.72, 32.64, 31.84, 19.57, 19.52, 19.48, 14.08; HRMS: calculated for C<sub>42</sub>H<sub>56</sub>O<sub>6</sub>Se<sub>2</sub>, 816.2402; found, 816.2393.

#### 2.5 Synthesis of 3a from 2a



The **2a** (0.216 g, 0.3 mmol) was dissolved in CH<sub>2</sub>Cl<sub>2</sub> (15 mL), and *tert*-butyl nitrite (TBN, 0.9 mmol) was introduced dropwisely. The resulting mixture was strirred at room temperature for 4 h. The solvent and the residue TBN was removed under reduced pressure to give the crude product, which was then purified by column chromatography on silica (eluent, CH<sub>2</sub>Cl<sub>2</sub>: Petro ether, 1:2, v/v) to give **3a** (orange-yellow solid, 0.204 g, 89%). mp: 40.8–42.2 °C; <sup>1</sup>H NMR (400 MHz, Chloroform-*d*):  $\delta$ (ppm) 7.60 (s, 1H), 4.50 (t, J = 6.5 Hz, 4H), 4.46-4.39 (m, 4H), 4.30 (t, J = 6.4 Hz, 2H), 4.19 (t, J = 6.4 Hz, 2H), 1.97-1.79 (m, 12H), 1.71-1.50 (m, 12H), 1.09-0.99 (m, 18H); <sup>13</sup>C NMR (100 MHz, Chloroform-*d*)  $\delta$ (ppm) 151.95, 146.82, 146.17, 145.29, 143.90, 142.45, 140.50, 132.75, 132.11,

128.19, 127.17, 125.19, 123.34, 122.63, 118.68, 113.40, 106.61, 75.85, 73.03, 72.93, 72.74, 72.63, 69.65, 32.47, 32.41, 32.39, 32.27, 31.52, 19.48, 19.34, 19.27, 19.17, 13.97, 13.94, 13.92; HRMS: calculated for C<sub>42</sub>H<sub>55</sub>NO<sub>8</sub>S<sub>2</sub>+ H<sup>+</sup>, 766.3442; found, 766.3436.

2.6 Synthesis of 3b from 2b



The **2b** (0.245 g, 0.3 mmol) was dissolved in CH<sub>2</sub>Cl<sub>2</sub> (15 mL), and TBN (0.9 mmol) was introduced dropwisely. The resulting mixture was strirred at room temperature for 4 h. The solvent and the residue TBN was removed under reduced pressure to give the crude product, which was then purified by column chromatography on silica (eluent, CH<sub>2</sub>Cl<sub>2</sub>: Petro ether, 1:2,  $\nu/\nu$ ) to give **3b** (yellow solid, 0.215 g, 81%). mp: 52.8–54.6 °C; <sup>1</sup>H NMR (400 MHz, Chloroform-*d*):  $\delta$ (ppm) 7.62 (s, 1H), 4.45-4.34 (m, 8H), 4.30 (t, *J* = 6.5 Hz, 2H), 4.19 (t, *J* = 6.3 Hz, 2H), 1.97-1.79 (m, 12H), 1.70-1.50 (m, 12H), 1.10-0.99 (m, 18H); <sup>13</sup>C NMR (100 MHz, Acetone-*d*<sub>6</sub>)  $\delta$ (ppm) 204.96, 151.11, 148.34, 146.97, 145.31, 141.84, 140.73, 129.83, 128.63, 128.55, 126.74, 123.01, 121.99, 119.72, 114.39, 105.78, 75.30, 72.52, 72.42, 72.26, 72.06, 68.89, 32.47, 32.39, 32.35, 32.33, 32.12, 31.35, 19.29, 19.23, 19.21, 19.16, 19.14, 18.97, 13.45, 13.41, 13.39, 13.35; HRMS: calculated for C4<sub>2</sub>H<sub>55</sub>NO<sub>8</sub>Se<sub>2</sub>+ H<sup>+</sup>, 862.2331; found, 826.2297.

2.7 Synthesis of 4a from 3a



Zn powder (0.325 g, 5 mmoL) was suspended in 5 mL absolute ethanol and 0.1 mL AcOH, then a solution of **3a** (0.765 g, 1mmoL) in 2 mL ethanol was added dropwise during a period of 10 min at 0

°C. The resulted mixture was stirred for 3 h at room temperature. The reaction mixture was filtered and the solvent of the collected filtrate was removed in *vacuo* to give crude product. The crude product was purified by column chromatography on silica (eluent, CH<sub>2</sub>Cl<sub>2</sub>: Petro ether, 1:2, v/v) to give **4a** (yellowish brown solid, 0.661g, 90%). For **4a**, mp: 43.3-45.3 °C; <sup>1</sup>H NMR (400 MHz, Chloroform*d*)  $\delta$ (ppm) 8.11 (s, 1H), 4.68 (s, 2H), 4.52-4.37 (m, 8H), 4.26 (t, J = 6.5 Hz, 4H), 4.17 (t, J = 6.7 Hz, 2H), 1.97-1.79 (m, 12H), 1.70-1.59 (m, 12H), 1.10-1.02 (m, 18H); <sup>13</sup>C NMR (100 MHz, Chloroform*d*):  $\delta$ (ppm) 151.53, 146.55, 146.07, 145.04, 142.59, 139.20, 138.05, 132.89, 127.52, 124.57, 120.75, 111.82, 109.95, 73.56, 72.95, 72.89, 72.77, 72.24, 70.72, 32.67, 32.48, 32.43, 32.42, 32.40, 31.86, 29.70, 19.49, 19.44, 19.33, 19.30, 14.04, 13.96; HRMS: calculated for C<sub>42</sub>H<sub>57</sub>NO<sub>6</sub>S<sub>2</sub>+ H<sup>+</sup>, 736.3700; found, 736.3713.

### 2.8 Synthesis of 4b from 3b



Zn powder (0.352g, 5.4 mmoL) was suspended in 5 mL absolute ethanol and 0.1 mL AcOH, then a solution of **3b** (0.932 g, 1.1mmoL) in 2 mL ethanol was added dropwise during a period of 10 min. at 0 °C. The resulted mixture was stirred for 3 h at room temperature. The reaction mixture was filtered and the solvent of the collected filtrate was removed in *vacuo* to give crude product. The crude product was purified by column chromatography on silica (eluent, CH<sub>2</sub>Cl<sub>2</sub>: Petro ether, 1:2,  $\nu/\nu$ ) to give **4b** (yellowish brown solid, 0.823g, 90% ). For **4b**, mp: 60.6-62.1°C; <sup>1</sup>H NMR (400 MHz, Chloroform-*d*):  $\delta$ (ppm) 8.32 (s, 1H), 4.65 (s, 2H), 4.42-4.32 (m, 8H), 4.26 (t, *J* = 6.5 Hz, 4H), 4.19 (t, *J* = 6.7 Hz, 2H), 1.98-1.82 (m, 12H), 1.71-1.56 (m, 12H), 1.10-1.00 (m, 18H); <sup>13</sup>C NMR (101 MHz, Chloroform-*d*):  $\delta$ (ppm) 151.03, 148.08, 147.12, 144.04, 139.55, 138.13, 132.46, 131.07, 126.36, 124.23, 120.32, 114.27, 109.18, 73.40, 73.01, 72.93, 72.72, 72.43, 69.79, 32.72, 32.66, 32.62, 32.60, 31.80, 19.52, 19.49, 19.46, 19.44, 19.42, 14.08, 14.02; HRMS: calculated for C4<sub>2</sub>H<sub>57</sub>NO<sub>6</sub>Se<sub>2</sub>+ H<sup>+</sup>, 832.2589; found, 832.2575.

### 2.9 Synthesis of 5a from 4a



The **4a** (0.073g, 0.1 mmol) and benzaldehyde (0.04ml, 0.2 mmol) were dissolved in TFA (3 mL) and sealed in a Schlenk tube. The resulting mixture was heated with stirring at the 120 °C for 12 h. After the reaction was finished, the reaction mixture was diluted with CH<sub>2</sub>Cl<sub>2</sub> and neutralized with NaHCO<sub>3</sub>, extracted with CH<sub>2</sub>Cl<sub>2</sub> (3 × 10 mL). The organic layer was dried with anhydrous Na<sub>2</sub>SO<sub>4</sub>, and the filtrate was evaporated under vacuum and purified by column chromatography on silica (eluent, CH<sub>2</sub>Cl<sub>2</sub>: Petro ether, 1:2,  $\nu/\nu$ ) to give the corresponding product **5a** (yellow solid, 0.058g, 71%). For **5a**, mp: 100.8-102.8°C; <sup>1</sup>H NMR (400 MHz, Chloroform-*d*):  $\delta$ (ppm) 7.97 (d, *J* = 5.8 Hz, 2H), 7.47-7.45 (m, 3H), 4.74-4.53 (m, 10H), 3.53 (t, *J* = 6.6 Hz, 2H), 2.08-1.91(m, 12H), 1.77-1.64 (m, 12H), 1.19-0.98 (m, 15H), 0.79 (t, *J* = 7.2 Hz, 3H); <sup>13</sup>C NMR (101 MHz, Chloroform-*d*):  $\delta$ (ppm) 158.04, 148.42, 148.22, 146.92, 146.81, 145.92, 145.67, 142.68, 135.57, 133.22, 130.58, 127.97, 127.00, 126.36, 126.05, 125.97, 125.35, 124.99, 122.20, 116.95, 112.34, 73.26, 73.04, 72.92, 72.80, 32.68, 32.51, 32.47, 32.39, 32.37, 31.24, 19.42, 19.37, 19.03, 14.04, 13.99, 13.91; HRMS: calculated for C4<sub>9</sub>H<sub>59</sub>NO<sub>6</sub>S<sub>2</sub>+ H<sup>+</sup>, 822.3858; found, 822.3845.

2.10 Synthesis of 5b from 4b



The **4b** (0.083g, 0.1 mmol) and phenylaldehyde (0.04 ml, 0.2 mmol) were dissolved in TFA (3 mL) and sealed in a Schlenk tube. The resulting mixture was heated with stirring at 120 °C for 12

hour. After the reaction was finished, the reaction mixture was diluted with CH<sub>2</sub>Cl<sub>2</sub> and neutralized with NaHCO<sub>3</sub>, extracted with CH<sub>2</sub>Cl<sub>2</sub> (3 × 10 mL). The organic layer was dried with anhydrous Na<sub>2</sub>SO<sub>4</sub>, and the filtrate was evaporated under vacuum and purified by column chromatography on silica (eluent, ethyl acetate: Petro ether, 1:25,  $\nu/\nu$ ) to give the corresponding product **5b** (yellow solid, 0.067g, 73%). For **5b**, mp: 137.5-139.3 °C; <sup>1</sup>H NMR (400 MHz, Chloroform-*d*):  $\delta$ (ppm) 7.98-7.92 (m, 2H), 7.58-7.51 (m, 3H), 4.72-4.45 (m, 10H), 3.62 (t, *J* = 6.7 Hz, 2H), 2.05-1.90(m, 12H), 1.78-1.64 (m, 12H), 1.14-1.01 (m, 15H), 0.82 (t, *J* = 7.1 Hz, 3H); <sup>13</sup>C NMR (100 MHz, Chloroform-*d*):  $\delta$ (ppm) 158.04, 149.94, 149.82, 149.02, 148.05, 147.42, 146.15, 143.03, 135.74, 133.61, 130.28, 129.67, 129.07, 128.89, 128.47, 127.84, 126.98, 123.41, 117.15, 113.55, 75.64, 75.37, 73.50, 73.14, 73.03, 72.73, 32.65, 32.61, 32.58, 32.50, 32.46, 31.25, 19.45, 19.44, 19.41, 19.05, 14.01, 14.00, 13.91; HRMS: calculated for C<sub>49</sub>H<sub>59</sub>NO<sub>6</sub>Se<sub>2</sub>+ H<sup>+</sup>, 918.2746; found, 918.2741.

### 2.11 Synthesis of 6a from 4a



The **4a** (0.073g, 0.1 mmol) and 2-pyridinecarboxaldehyde (0.038 ml, 0.2 mmol) were dissolved in TFA (3 mL) and sealed in a Schlenk tube. The resulting mixture was heated with stirring at 120 °C for 12 h. After the reaction was finished, the reaction mixture was diluted with CH<sub>2</sub>Cl<sub>2</sub> and neutralized with NaHCO<sub>3</sub>, extracted with CH<sub>2</sub>Cl<sub>2</sub> (3 × 10 mL). The organic layer was dried with anhydrous Na<sub>2</sub>SO<sub>4</sub>, and the filtrate was evaporated under vacuum and purified by column chromatography on silica (eluent, petro ether: ethyl acetate, 8 : 1,  $\nu/\nu$ ) to give the corresponding product **6a** (yellow solid, 0.066g, 80%). For **6a**, mp: 126.7-128.4 °C; <sup>1</sup>H NMR (400 MHz, Methylene Chloride-*d*<sub>2</sub>):  $\delta$ (ppm) 8.74 (d, *J* = 4.8, 1H), 7.91 (t, *J* = 6.8 Hz, 1H), 7.84 (d, *J* = 7.6 Hz, 1H), 7.49-7.40(m, 1H), 4.63-4.49 (m, 10H), 3.79 (t, *J* = 6.8 Hz, 2H), 1.98-1.84 (m, 10H), 1.72-1.54 (m, 12H), 1.25-1.13 (m, 2H), 1.08-0.99 (m, 12H), 0.95 (t, *J* = 7.4 Hz, 3H), 0.82 (t, *J* = 7.3 Hz, 3H); <sup>13</sup>C NMR (100 MHz, Methylene Chloride-*d*<sub>2</sub>):  $\delta$ (ppm) 161.19, 156.92, 148.76, 148.34, 148.31, 146.55, 145.54, 135.66, 135.22, 133.27, 129.19, 125.83, 125.80, 125.05, 124.12, 122.65, 121.51, 117.29, 112.29, 75.57, 74.42, 73.10, 72.93, 72.77, 32.74, 32.62, 32.57, 32.50, 32.49, 31.82, 19.50, 19.48, 19.45, 19.16, 13.93, 13.88, 13.86; HRMS: calculated for C<sub>48</sub>H<sub>58</sub>N<sub>2</sub>O<sub>6</sub>S<sub>2</sub>+ H<sup>+</sup>, 823.3809; found, 823.3821.

2.12 Synthesis of 6b from 4b



The **4b** (0.083g, 0.1 mmol) and 2-pyridinecarboxaldehyde (0.038 ml, 0.2 mmol) were dissolved in TFA (3 mL) and sealed in a Schlenk tube. The resulting mixture was heated with stirring at 120 °C for 12 h. After the reaction was finished, the reaction mixture was diluted with CH<sub>2</sub>Cl<sub>2</sub> and neutralized with NaHCO<sub>3</sub>, extracted with CH<sub>2</sub>Cl<sub>2</sub> (3 × 10 mL). The organic layer was dried with anhydrous Na<sub>2</sub>SO<sub>4</sub>, and the filtrate was evaporated under vacuum and purified by column chromatography on silica (eluent, Petro ether: ethyl acetate, 15:1,  $\nu/\nu$ ) to give the corresponding product **6b** (yellow solid, 0.058g, 80% ). For **6b**, mp: 103.4-104.1 °C; <sup>1</sup>H NMR (400 MHz, Chloroform-*d*):  $\delta$ (ppm) 8.78 (d, *J* = 4.7, 1H), 7.95-7.84 (m, 2H), 7.48-7.42 (m, 1H), 4.65-4.45 (m, 10H), 3.86 (t, *J* = 6.8 Hz, 2H), 2.01-1.87 (m, 10H), 1.75-1.59 (m, 12H), 1.25-1.16 (m, 2H), 1.04-1.01(m, 12H), 0.97 (t, *J* = 7.4 Hz, 3H), 0.85 (t, *J* = 7.3 Hz, 3H); <sup>13</sup>C NMR (100 MHz, Chloroform-*d*):  $\delta$ (ppm) 161.20, 156.57, 150.07, 149.75, 148.09, 147.26, 146.30, 135.67, 135.53, 133.93, 129.64, 128.32, 127.05, 125.77, 125.44, 124.01, 123.23, 122.43, 117.40, 75.71, 74.44, 73.50, 73.08, 72.99, 72.84, 32.57, 32.55, 32.53, 32.47, 32.44, 31.66, 19.42, 19.30, 19.01, 14.01, 13.98, 13.97, 13.92; HRMS: calculated for C<sub>48</sub>H<sub>58</sub>N<sub>2</sub>O<sub>6</sub>Se<sub>2</sub>+ H<sup>+</sup>, 919.2698; found, 919.2696.

# III. Thermogravimetric Analyses (TGA)

The thermogravimetric analyses (TGA) of compounds **5a–6b** were conducted on 1090B type thermal analyzer(Dupont Engineering Polymers), and Table S1 summarizes the degradation temperature ( $T_d$ ) of the compounds.

Table S1. Thermal stability of the representative compounds.

Compounds	5a	6a	5b	6b
<i>T</i> d [°C]	311	304	322	288



Figure S1. Thermogravimetric analyses of compounds 5a-6b

# **IV. Crystal Structure Analysis**

	6a	6b	6a[ZnCl <sub>2</sub> ]	6b[ZnCl <sub>2</sub> ]
CCDC number	1830627	1830628	1830629	1830630
Empirical formula	$C_{48}H_{58}N_2O_6S_2$	$C_{48}H_{58}N_2O_6Se_2$	$C_{48}H_{58}Cl_2N_2O_6S_2Zn \\$	$C_{48}H_{58}Cl_2N_2O_6Se_2Zn$
Formula weight	823.08	916.88	959.35	1053.15
Temperature [K]	150(2)	150(2)	150(2)	150(2)
Apparatus	Bruker APEX-II CCD	Bruker APEX-II CCD	Bruker APEX-II CCD	Bruker APEX-II CCD
Radiation	Cu-Ka	Cu-Ka	Cu-Ka	Cu-Ka
λ[Å]	1.54178	1.54178	1.54178	1.54178
Crystal size [mm <sup>3</sup> ]	0.25×0.14×0.09	0.30×0.10×0.09	0.35×0.13×0.09	0.39×0.15×0.09
Crystal shape	needle	needle	needle	needle
Crystal color	pale yellow	pale yellow	red	red
Crystal system	Monoclinic	Monoclinic	Monoclinic	Monoclinic
Space group	<i>P</i> 2 <sub>1</sub> / <i>c</i> (#14)	<i>P</i> 2 <sub>1</sub> / <i>c</i> (#14)	<i>P</i> 2 <sub>1</sub> / <i>c</i> (#14)	$P2_1/c$ (#14)
<i>a</i> [Å]	7.9035(2)	7.8970(3)	15.7813(2)	15.4628(6)
<i>b</i> [Å]	17.3632(4)	17.4785(6)	32.2684(15)	32.3858(13)
<i>c</i> [Å]	30.8922(10)	31.1201(10)	9.0074(5)	9.1810(4)
α [°]	90	90	90	90
β [°]	93.818(2)	92.646(1)	97.563(2)	93.890(2)
γ [°]	90	90	90	90
V[Å <sup>3</sup> ]	4229.9(2)	4290.9(3)	4547.0(4)	4587.0(3)
Z	4	4	4	4
$d_{\rm cal}$ [g cm <sup>-3</sup> ]	1.292	1.419	1.401	1.525
$\mu [\mathrm{mm}^{-1}]$	1.557	2.566	3.099	4.037
$2 heta_{\max}$ [°]	144.876	135.358	136.886	133.434
Data/restraints/parameter	8309/0/529	8402/0/523	8295/0/556	8073/302/645
GooF	1.061	1.089	1.065	1.048
$R (I > 2\sigma I)$	0.036	0.035	0.049	0.090
$wR_2$	0.0878	0.0891	0.1268	0.2253

Table S2. Selected crystallographic data for 6a, 6b, 6a[ZnCl<sub>2</sub>], and 6b[ZnCl<sub>2</sub>]



**Figure S2**. Crystal structure of **6a**: a) top and b) side view the molecule; c) packing structures. The pale cyan, grey, blue, red, and olive balls represent hydrogen, carbon, nitrogen, oxygen, and sulfur atoms, respectively.



**Figure S3**. Crystal structure of **6b**: a) top and b) side view the molecule; c) packing structures. The pale cyan, grey, blue, red, and orange balls represent hydrogen, carbon, nitrogen, oxygen, and selenium atoms, respectively.



**Figure S4**. Comparison of selected bond lengths in the crystal structures of **6a** and **6a**[**ZnCl**<sub>2</sub>]. The pale cyan, grey, blue, red, olive, green, and blue grey balls represent hydrogen, carbon, nitrogen, oxygen, sulfur, chlorine, and zinc atoms, respectively.



**Figure S5**. Comparison of selected bond lengths in the crystal structures of **6b** and **6b[ZnCl<sub>2</sub>]**. The pale cyan, grey, blue, red, olive, orange, and blue grey balls represent hydrogen, carbon, nitrogen, oxygen, selenium, chlorine, and zinc atoms, respectively.

# V. Theoretical calculation

All calculations were carried out with the Gaussian 09 programs.<sup>[S4]</sup> The molecules were optimized the energy levels of HOMO and LUMO were calculated with the B3LYP/6-311G(d,p). Nucleus independent chemical shifts (NICS) were calculated with the Gaussian 09 software using the gauge invariant atomic orbital (GIAO) approach at the GIAO-B3LYP/6-311+G(d,p) level.





Compounds	1 ring	2 ring	3 ring	4 ring	5 ring	6 ring	7 ring	8 ring
6a	-13.01	-0.83	-12.85	-12.40	-12.27	-12.63	-4.22	-6.00
6a[H <sup>+</sup> ]	-13.46	-1.80	-13.38	-11.36	-10.22	-9.80	-5.01	-5.60
6a[H <sup>+</sup> ]2	-13.74	-3.00	-13.16	-11.15	-12.28	-10.67	-6.25	-6.51
6a[ZnCl <sub>2</sub> ]	-13.19	-0.76	-12.68	-12.17	-8.41	-11.04	-4.67	-5.85
6b	-12.13	-0.59	-12.40	-12.17	-9.99	-10.46	-4.21	-6.15
6b[H <sup>+</sup> ]	-12.67	-1.58	-13.08	-11.18	-9.71	-8.74	-4.95	-5.64
6b[H <sup>+</sup> ] <sub>2</sub>	-13.05	-2.58	-12.75	-10.66	-12.16	-13.04	-5.90	-6.53
6b[ZnCl <sub>2</sub> ]	-12.50	-0.74	-12.33	-12.07	-8.83	-12.75	-4.66	-5.97

Compounds	Energy levels / eV							
Compounds	HOMO-1	НОМО	LUMO	LUMO+1	Eg <sup>[a]</sup>			
6a	-5.64	-5.57	-2.11	-1.37	3.46			
6a[H <sup>+</sup> ]	-8.59	-8.22	-5.91	-4.51	2.31			
6a[H <sup>+</sup> ] <sub>2</sub>	-10.66	-10.40	-9.01	-8.21	1.39			
6a[ZnCl <sub>2</sub> ]	-6.19	-5.98	-3.00	-1.98	2.98			
6b	-5.59	-5.47	-2.06	-1.33	3.41			
6b[H <sup>+</sup> ]	-8.48	-8.15	-5.85	-4.43	2.30			
6b[H <sup>+</sup> ] <sub>2</sub>	-10.54	-10.32	-8.92	-8.15	1.40			
6b[ZnCl <sub>2</sub> ]	-6.11	-5.93	-2.97	-1.93	2.96			

Table S4. The calculated energy level for the frontier orbitals for the representative compounds.

<sup>[a]</sup> $E_{\rm g} = E_{\rm LUMO} - E_{\rm HOMO}$ 



Figure S6. Schematic plot of HOMO–LUMO levels of the representative compounds.



Figure S7. Calculated molecular orbitals of compound 6a.



Figure S8. Calculated molecular orbitals of compound  $6a[H^+]$ .



Figure S9. Calculated molecular orbitals of compound  $6a[H^+]_2$ 



Figure S10. Calculated molecular orbitals of compound 6a[ZnCl<sub>2</sub>]



Figure S11. Calculated molecular orbitals of compound 6b.



Figure S12. Calculated molecular orbitals of compound  $6b[H^+]$ .



Figure S13. Calculated molecular orbitals of compound  $6b[H^+]_2$ 



Figure S14. Calculated molecular orbitals of compound  $6b[ZnCl_2]$ 

# **VI. Photophysical Study**

## (1) UV-Vis Spectra

The UV-Vis spectra of the compounds so far obtained were measured in their dichloromethane (CH<sub>2</sub>Cl<sub>2</sub>) solution ( $c = 1.0 \times 10^{-5} \text{ mol L}^{-1}$ ) at 20 °C on a UV-2600 UV-Vis spectrometer (Shimadzu). Table S5 summarizes the absorption spectra data of the compounds.

Comp.	$\lambda_{max}/nm$	log ε	$\lambda_{max}/nm$	log ε	$\lambda_{max}/nm$	$\log \varepsilon$	$\lambda_{max}/nm$	$\log \varepsilon$	$\lambda_{max}/nm$	log ε
5a	296	4.56	325	4.64	337	4.61	404	4.11	427	4.01
6a	295	4.63	325	4.70	337	4.68	404	4.18	426	4.08
5b	295	4.64	325	4.55	340	4.51	405	4.01		
6b	294	4.68	324	4.73	336	4.69	403	4.20	426	4.11

Table S5. UV/Vis spectra of compounds 5a–6b inCH<sub>2</sub>Cl<sub>2</sub> solution <sup>[a]</sup>.



Figure S15. UV-Vis absorption spectra of compounds in  $CH_2Cl_2$  solution (10<sup>-5</sup> mol L<sup>-1</sup>) at 20 °C.



**Figure S16**. UV-Vis absorption spectra of **5b** in  $CH_2Cl_2$  (10<sup>-5</sup> mol L<sup>-1</sup>) upon titration with TFA and neutralization with Et<sub>3</sub>N.



**Figure S17**. UV-Vis absorption spectra of **6b** in  $CH_2Cl_2$  (10<sup>-5</sup> mol L<sup>-1</sup>) upon titration with TFA and neutralization with Et<sub>3</sub>N.



Figure S18. UV-Vis absorption spectra of 6b in  $CH_2Cl_2$  (10<sup>-5</sup> mol L<sup>-1</sup>) upon titration with ZnCl<sub>2</sub>.

### (2) Fluorescence

Fluorescence excitation and emission spectra were recorded with an RF-5301(pc)s Spectrofluorophotometer, fluorescence lifetime and steady state were measured on FLS920 Spectrofluorophotometer. Measurement conditions: solvent, CH<sub>2</sub>Cl<sub>2</sub>; concentration,  $10^{-5}$  mol L<sup>-1</sup>; temperature, 20 °C.

	$\lambda_{\mathrm{ex}}$ / nm	$\lambda_{\rm em}$ / nm	Stocks shift / cm <sup>-1</sup>	$oldsymbol{\Phi}_{\mathrm{F}}$ / %	$ au_1$ / ns
5a	439	493	2658	3.5	4.2
6a	439	512	3248	4.2	3.5
5b	432	483	2444	[b]	[b]
6b	433	496	2933	_[b]	_[b]

Table S6. The emission and excitation properties of compounds 5a–6b in CH<sub>2</sub>Cl<sub>2</sub> solution.<sup>[a]</sup>

<sup>[a]</sup>  $\lambda_{ex}$ : excitation wavelength;  $\lambda_{em}$ : maximum emission wavelength;  $\Phi_F$ : fluorescence quantum yield;  $\tau_1$  fluorescence lifetime. [b] the fluorescence quantum yield and lifetime cannot be defined due to the weak emissions in CH<sub>2</sub>Cl<sub>2</sub>.



Figure S19. Emission spectra of compounds in CH<sub>2</sub>Cl<sub>2</sub> solution ( $10^{-5}$  mol L<sup>-1</sup>) at 20 °C.



Figure S20. Emission spectra of  $6a[ZnCl_2]$  in the different solvents ( $c = 10^{-5} \text{ mol } L^{-1}$ ) at 20 °C, excitation wavelength 492 nm.

# VII. <sup>1</sup>H NMR evidence for coordination



Figure S21. Comparison of <sup>1</sup>H NMR spectra for 6a and 6a[ZnCl<sub>2</sub>] in CDCl<sub>3</sub>.



Figure S22. Comparison of <sup>1</sup>H NMR spectra for 6b and 6b[ZnCl<sub>2</sub>] in CDCl<sub>3</sub>.

# VIII.<sup>1</sup>H NMR (CDCl<sub>3</sub>), <sup>13</sup>C NMR (CDCl<sub>3</sub>) and IR Spectra of products





















<u>210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0</u>







 $-8.33 \\ +4.465 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.33 \\ +4.$ 





 $\begin{array}{c} 7.98 \\ 7.97 \\ 7.96 \\ 7.96 \\ 7.55 \\ 7.54 \\ 7.57 \\ 7.57 \end{array}$ 

# 







7.957.95 7.547.537.53-7.27

#### 

 $\begin{array}{c} 1.06 \\ 1.04 \\ 1.02 \\ 0.03 \end{array}$ 











88, 39 88, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87, 37 87



