Supporting Information

for

Cu₂Se, Ag₂Se, and AgCuSe Nanocrystals from Thermolysis

of Single-Source Metallomesogen Templates

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Figure S1. FTIR spectra taken of solid $AgSeC_{12}H_{25}$ and $CuSeC_{12}H_{25}$ precursor compounds. The *n*-alkane backbone follows expectations, which are well established in literature. The C-Se-M stretching vibration is shown at 639 cm⁻¹ for the Cu analog, and 637 cm⁻¹ for the Ag analog.



Figure S2. Additional TEM images taken from side-on vantage of $Cu_{1.8}$ Se nanoribbon structures. The calculated average thickness from 30 measurements is 2.30 +/- 0.25 nm. As the $Cu_{1.8}$ Se cubic structure has a=b=c=0.5767 nm, this thickness correlates with exactly 4 unit cells (expected 2.3068 nm). Panel a) shows the side-on TEM images of the nanoribbon thickness (left), and a zoomed-out image of the nanoribbon sample for added perspective, with a twisted ribbon shown (right). Panel b) shows time-dependent XRD of $CuSeC_{12}H_{25}$ decomposition taken from lower angle than shown in Figure 3. While for higher thermolysis durations we only run from $2\theta=7.5^{\circ}$, the spectra indicate a loss of bilayer spacing parameter with time. At 6 hours, most ribbons have lost stacked registry and so no significant peaks are observed here.



Figure S3. Ag₂Se spherical particles from AgSeC₁₂H₂₅ at 185 °C. Particles show vaguely hexagonal faceting and align along these facets to produce dimers, trimers, etc. with some examples highlighted here in red.



Figure S4. Ag₂Se nanorods synthesized from AgSeC₁₂H₂₅ at 150 °C which exhibit a propensity to self-assemble into closed packed arrays. We believe the TEM imaging indicates that these nanorods are aligned along their long axis. Interestingly, these rods show cubic cross section. However, it is not clear if these rod arrays are indicative of the entire sample. Possibly, these nanorods with cubic cross section simply account for one crystallographic phase, e.g. the population of NCs with a cubic phase. However, the measured edge length of these NCs is 6.8 \pm 0.6 nm which is in close agreement with measurements for the rod populations lying on their sides.



Figure S5. Polarized optical microscope images of $Ag_{0.5}Cu_{0.5}SeC_{12}H_{25}$. Top row images at 150 °C after 3, 10, 20, and 25 minutes on the heating stage (a-d); bottom row images at 145 °C after 3, 20, 30, and 35 minutes (e-h). These images demonstrate the decreased stability of the mixed-metal dodecaneselenolate, which undergoes slow isotropic transition at a lower temperature than is observed for either $AgSeC_{12}H_{25}$ or $CuSeC_{12}H_{25}$ individually.



Figure S6. EDX of metal selenide thermolysis products. All precursors are reacted for the specified parameters prior to washing. The inorganic residues are analyzed by EDX (average of dried NC aggregates at 3 locations). a-c) show results for $CuSeC_{12}H_{25}$, $AgSeC_{12}H_{25}$, and $Ag_{0.5}Cu_{0.5}SeC_{12}H_{25}$ respectively.



Figure S7. High resolution XRD scan of $AgSeC_{12}H_{25}$. The higher 20 peaks shown here are indicative of the in-plane Ag-Se bonding network. Peaks are indexed against Ag-alkanethiolates as reported by Dance et al (ref. #9). Although we stop short of definitively indexing the peaks, there is good agreement between the Ag-Se bonding network observed here and the Ag-S bonding network previously reported. We believe the compounds are structurally analogous to previously reported Ag-alkanethiolates.