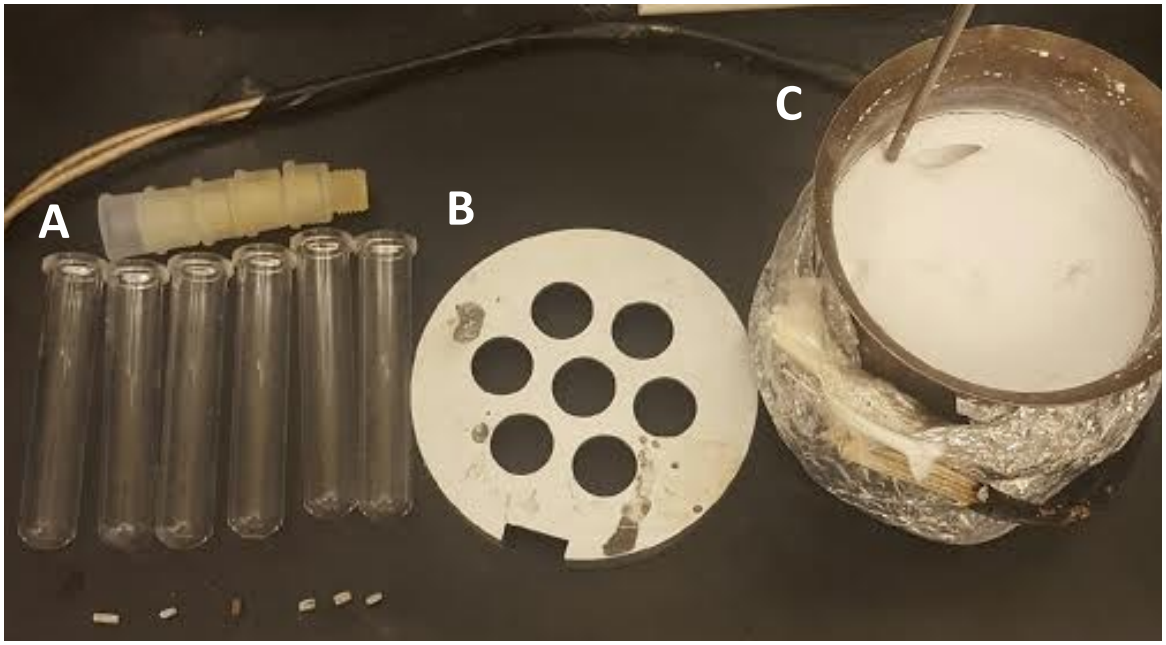
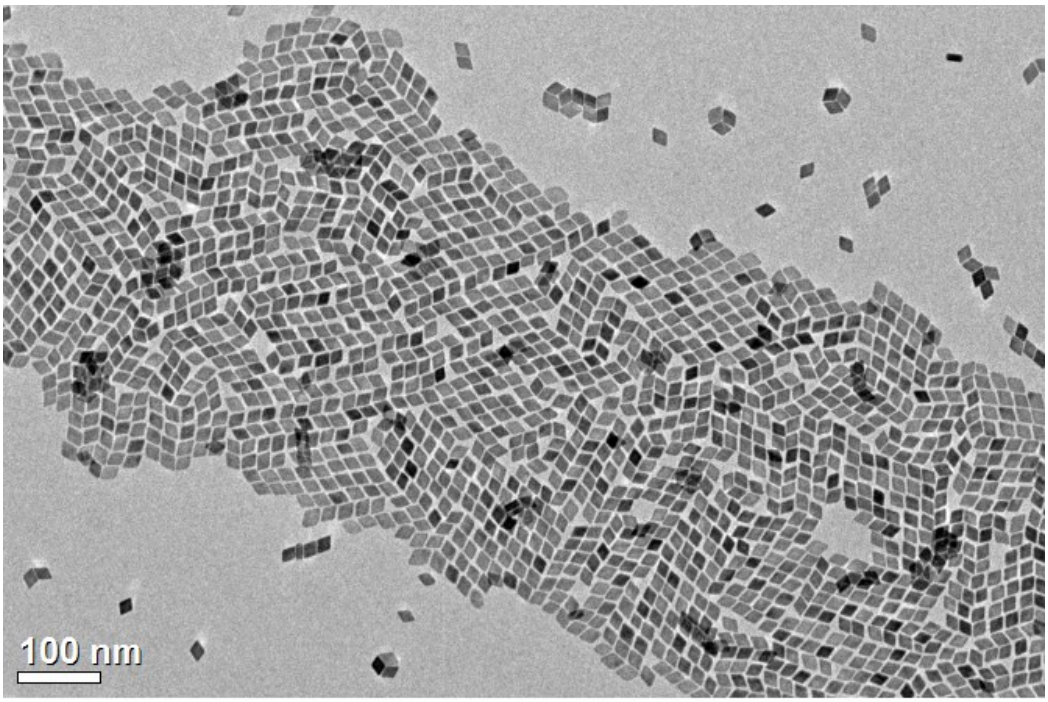
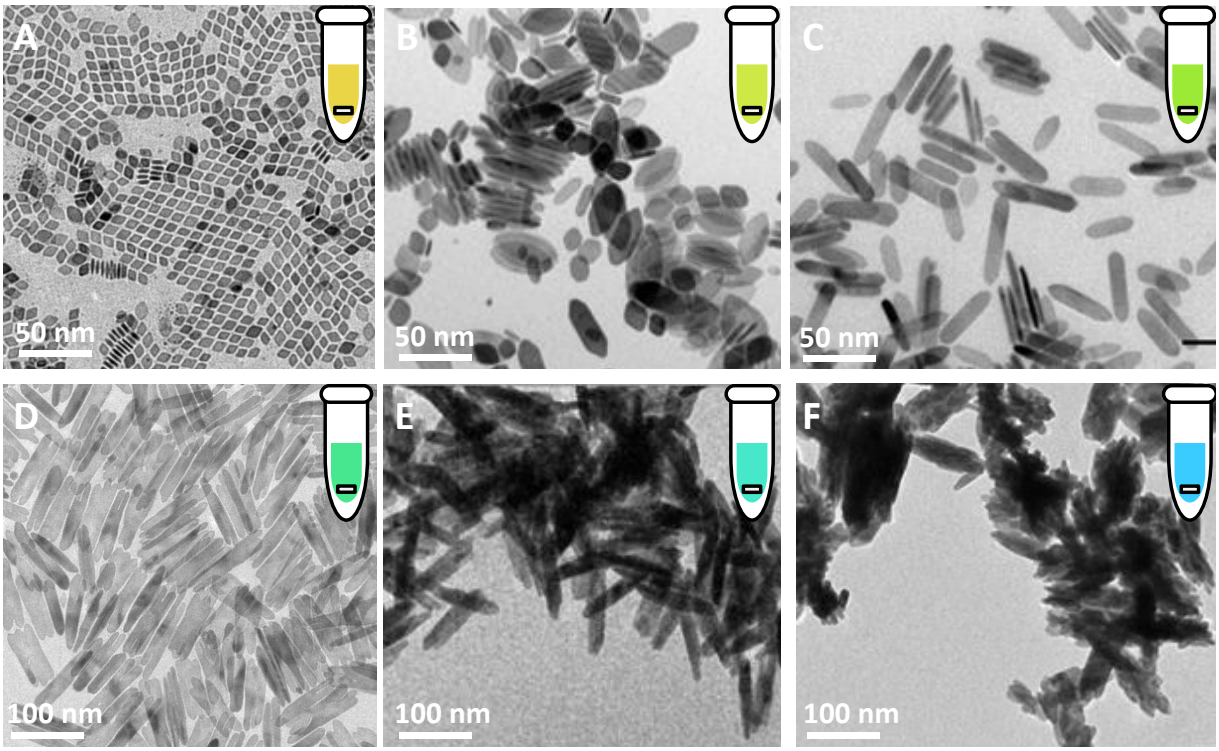
Supplemental Information:



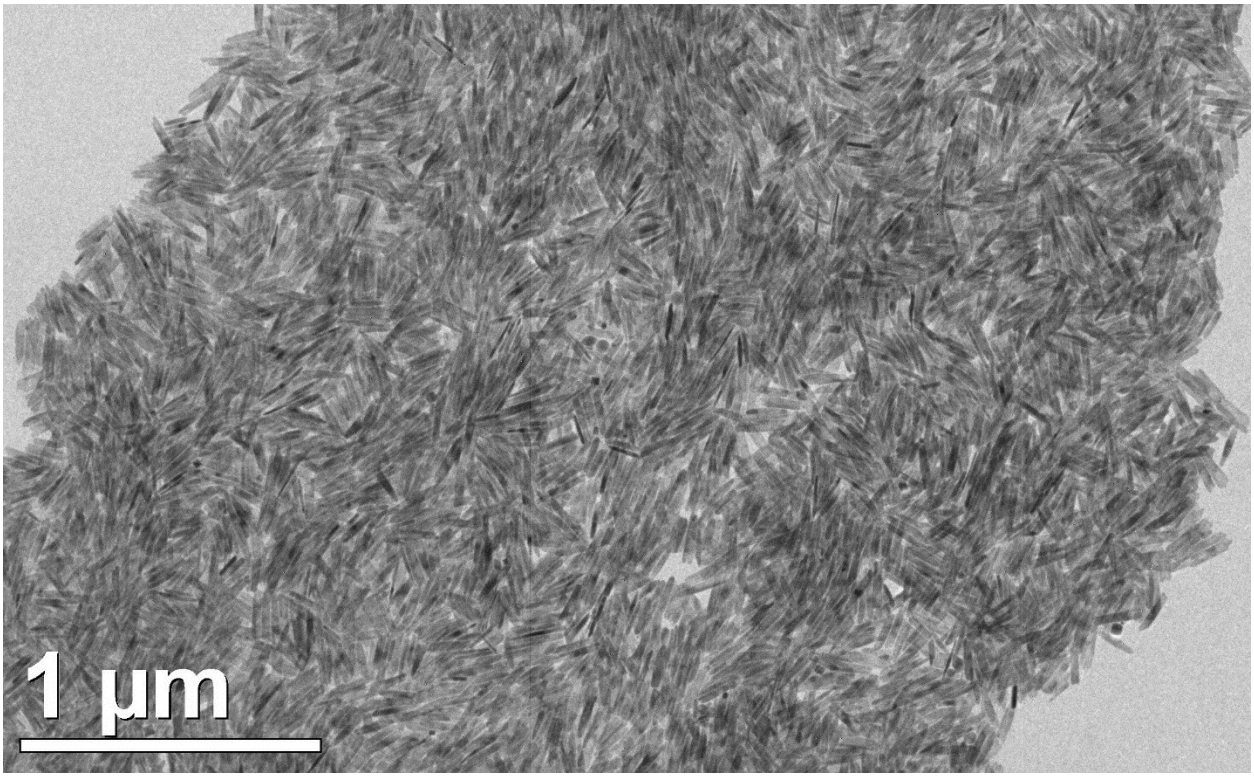
Supplementary Figure S1: Deconstructed microreactor setup. A) Borosilicate test tubes with silicone septa and micro stir bars. B) Aluminum test tube holder with hole dimensions designed to support a microreactor from the lip of its opening. C) Cooled salt bath containing a 1:1 mixture of KNO_3 to $NaNO_3$. The salt bath is contained in a stainless steel beaker that is wrapped in heat tape graded for 1000 ºC. Aluminum foil holds the tape in place. When hot, the molten salt is stirred by a magnetic stir bar.



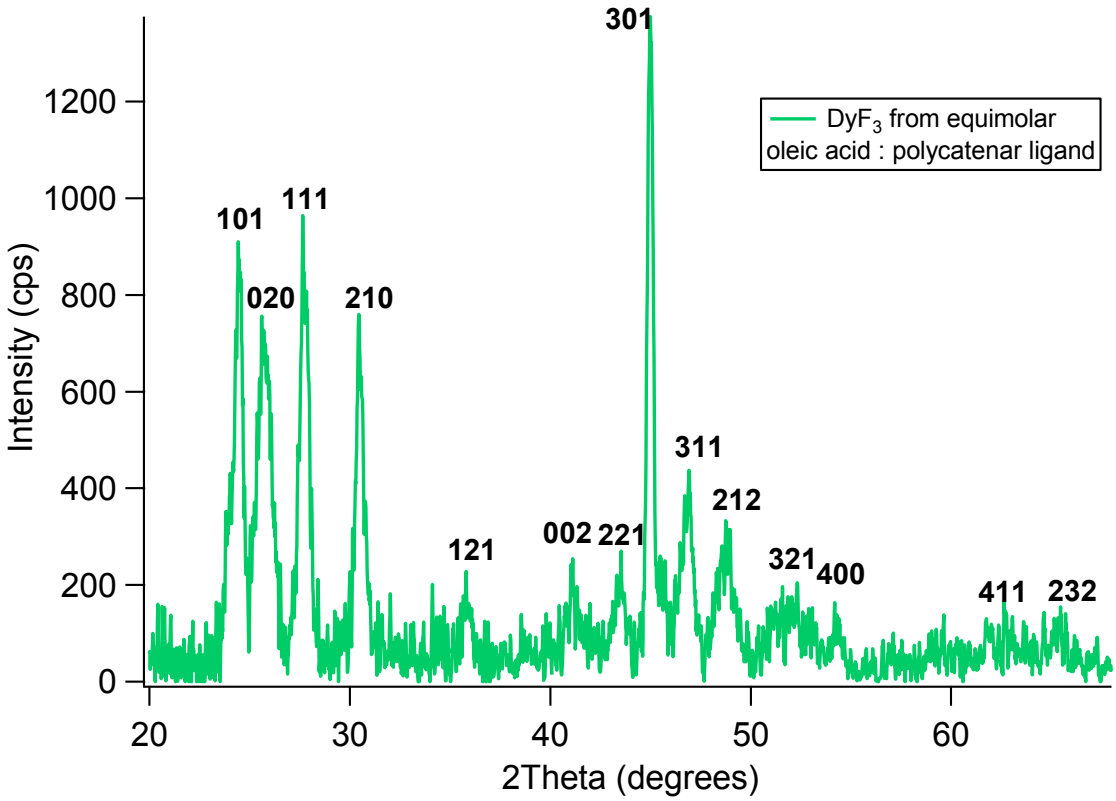
Supplemental Figure S2: Low-magnification TEM image of the DyF_3 rhombic plates obtained using the standard 1:1 oleic acid/octadecene reaction environment in the microreaction setup.



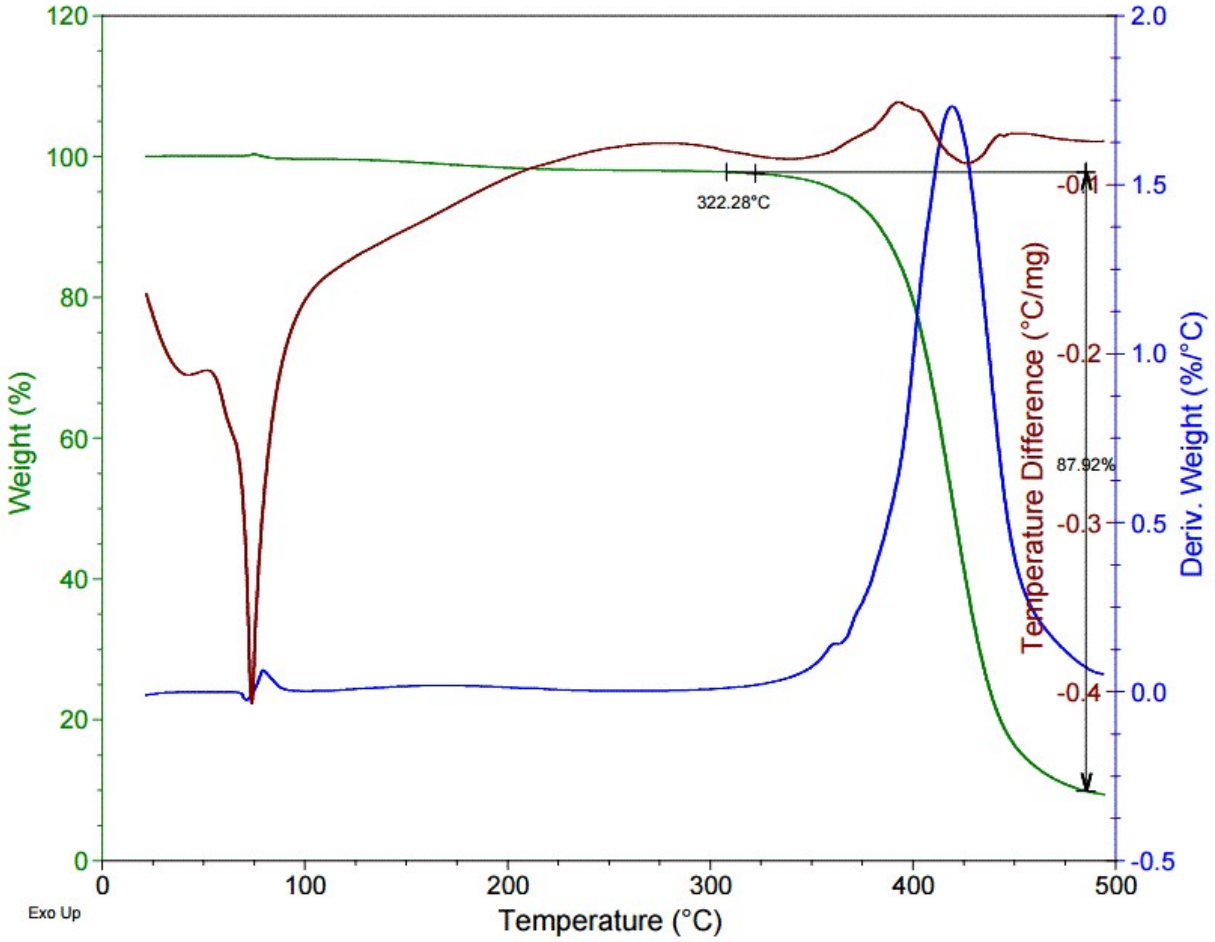
Supplementary Figure S3: Resulting DyF_3 nanoparticles by % molar replacement of oleic acid by the polycatenar ligands. A=0%, B=20%, C=40%, D=50%, E=80%, and F=100% molar replacement.



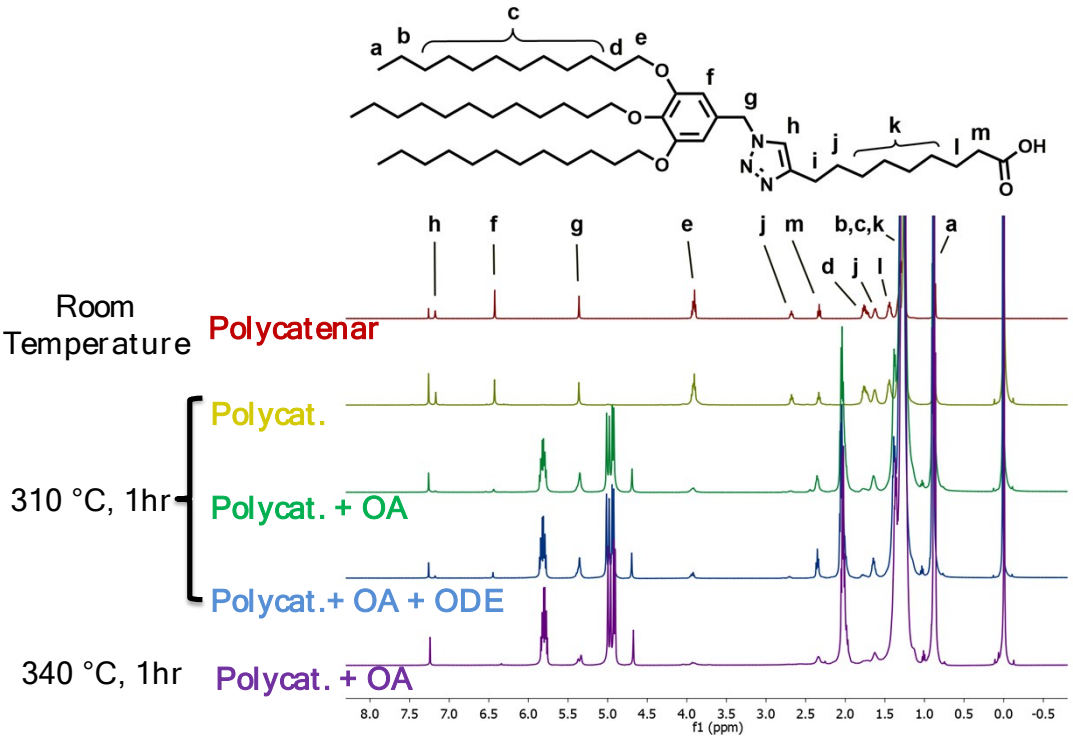
Supplemental Figure S4: Low-magnification image of the elongated plates achieved at a 50% molar replacement of oleic acid with the polycatenar ligands from the microreaction setup.



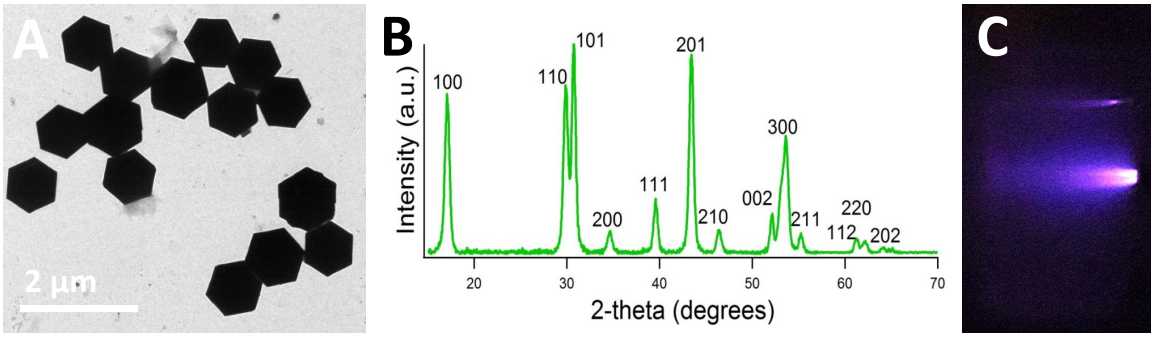
Supplemental Figure S5: Powder X-Ray diffraction pattern of the polycatenar DyF_3 trials. The peak assignment is indicative of the α-phase DyF_3



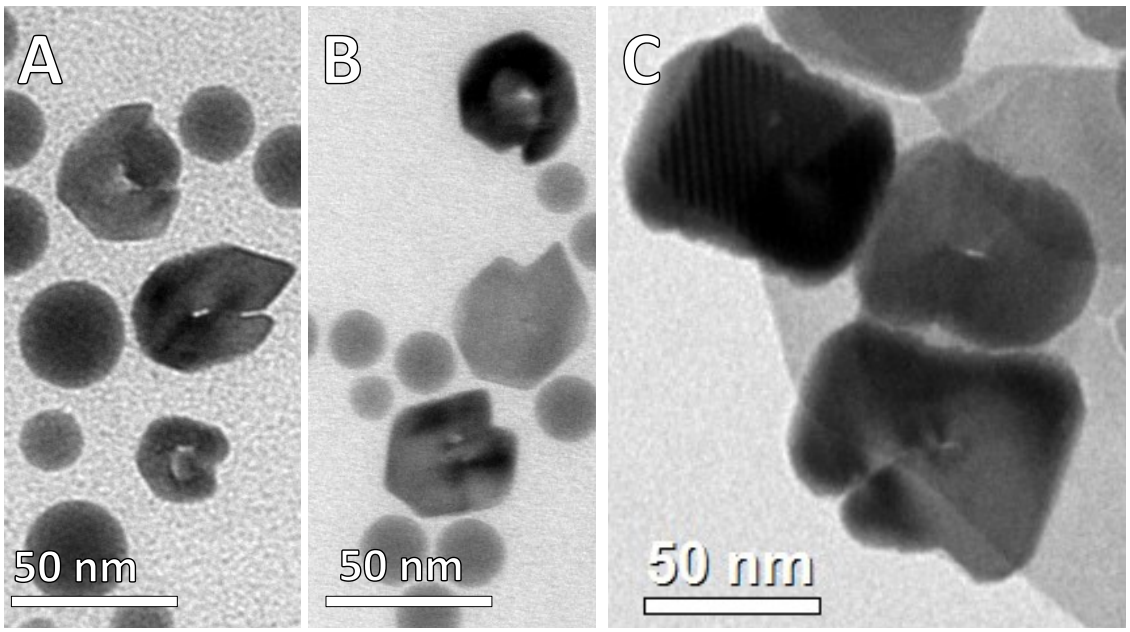
Supplemental Figure S6: The thermogravimetric analysis of the polycatenar ligand. Tracking the weight change as a function of temperature (green curve) indicates that decomposition of the ligand begins at 320 °C, which is above the temperature of the reaction.



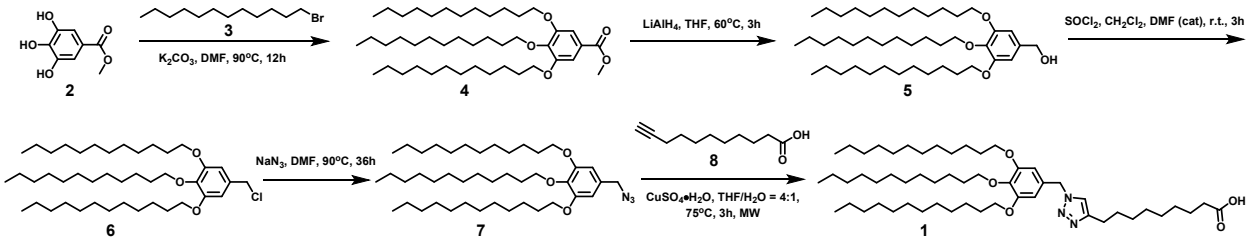
Supplemental Figure S7: NMR spectra of the polycatenar ligands and reaction mixtures after being held at the reported time for the noted amount of time. The results at 310 °C indicate that the ligand does not decompose during the synthesis. At 340 °C, the change in the peaks correlated to e, f, and m indicates the degradation of the ligand.



Supplemental Figure S8: β-$NaYF_4$: 0.2%Tm, 20%Yb upconverting nanocrystals synthesized from the microreactor vessel at 340°C for 40 min. A) Transmission electron microscopy confirms the expected hexagonal prism morphology. B) Powder x-ray diffraction with β-$NaYF_4$ peak assignment. C) Optical response upon 980nm excitation.



Supplemental Figure S9: TEM images of screw-dislocated and fused particles over time, at A) 10 min, B) 20 min, and C) 40 min.



Supplemental Scheme S1. Synthesis of polycatenar ligand **1**.