Electronic Supplementary Material (ESI) for Nanoscale.

SUPPORTING INFORMATION

Alkyl Selenol Reactivity with Common Solvents and Ligands: Influences on Phase Control in Nanocrystal Synthesis

Eric A. Ho, Antony R. Peng and Janet E. Macdonald

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Table S1: NMR parameters used for ^{1}H and ^{77}Se experiments.

	^{1}H-NMR	^{77}Se-NMR	^{77}Se-NMR (for upfield detection of TOP:Se)
Number of Scans	16	128	128
Spectral Width (ppm)	13	1000	1000
Recycle Delay (s)	-	5	5
Centre Shift (ppm)	5	250	-500

^{1}H NMR of all DDSeH and ligand combinations at 25 °C, 155 °C and 220 °C.

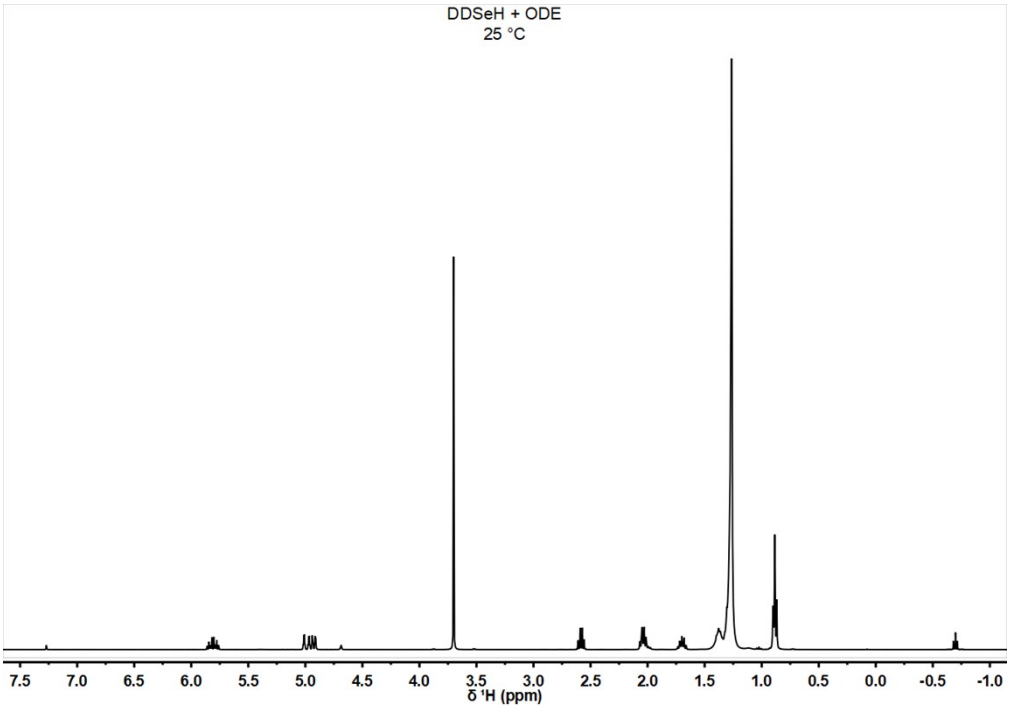

Figure S1: ^{1}H NMR of DDSeH and ODE at 25 °C in $CDCl_3$. Dioxane internal standard at δ = 3.71 ppm

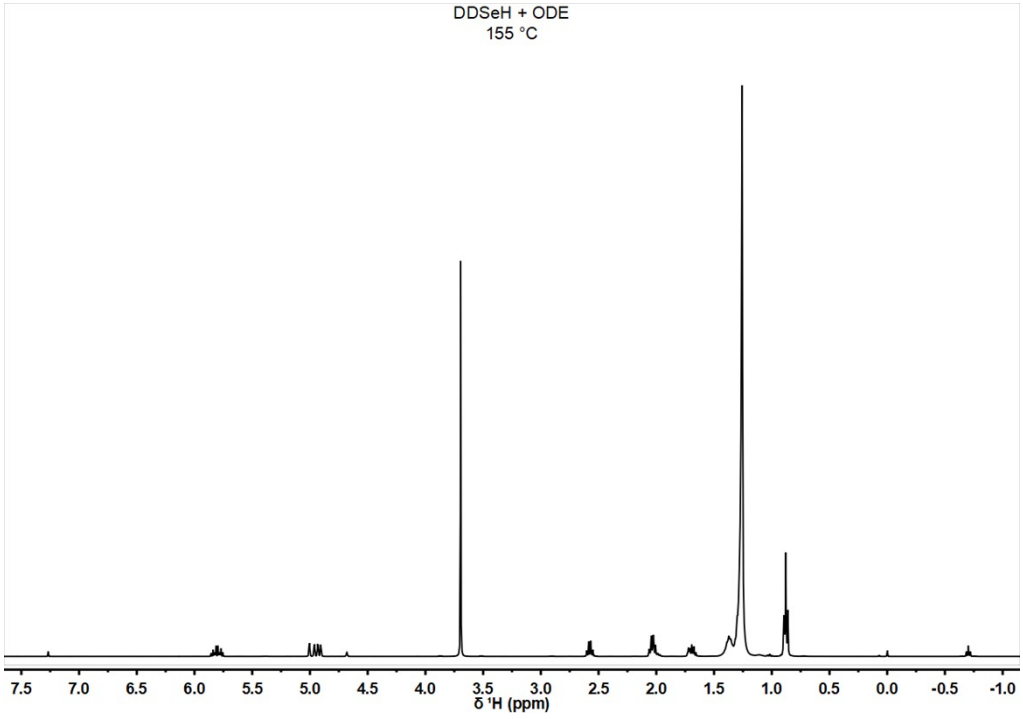

Figure S2: ^{1}H NMR of DDSeH and ODE at 155 °C in $CDCl_3$. Dioxane internal standard at δ = 3.71 ppm

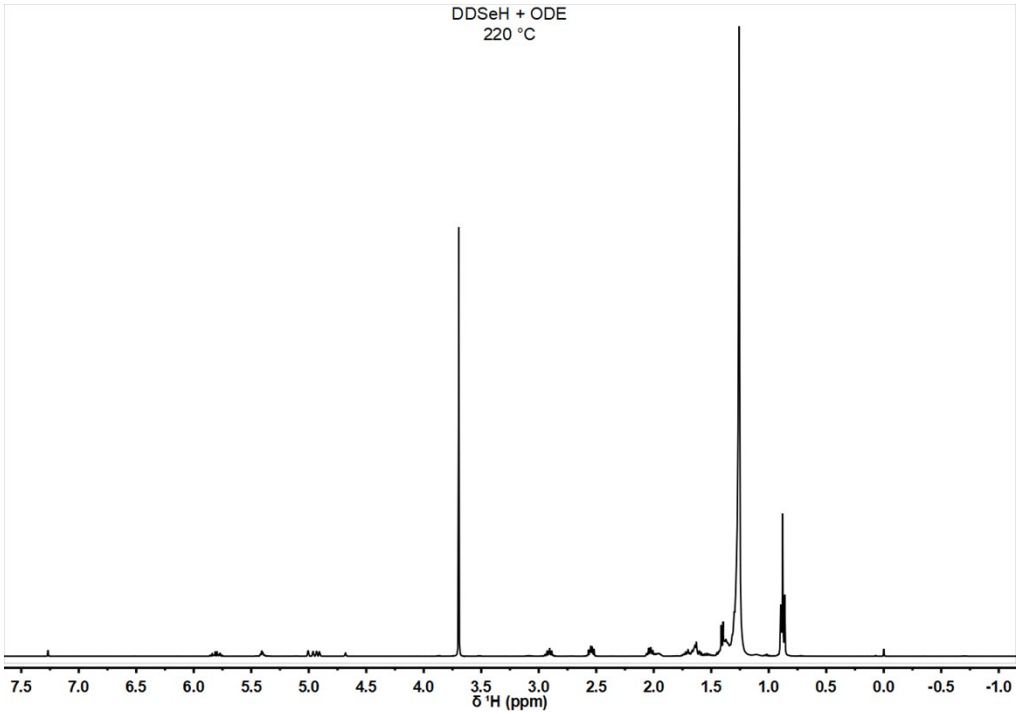

Figure S3: ^{1}H NMR of DDSeH and ODE at 220 °C in $CDCl_3$. Dioxane internal standard at δ = 3.71 ppm

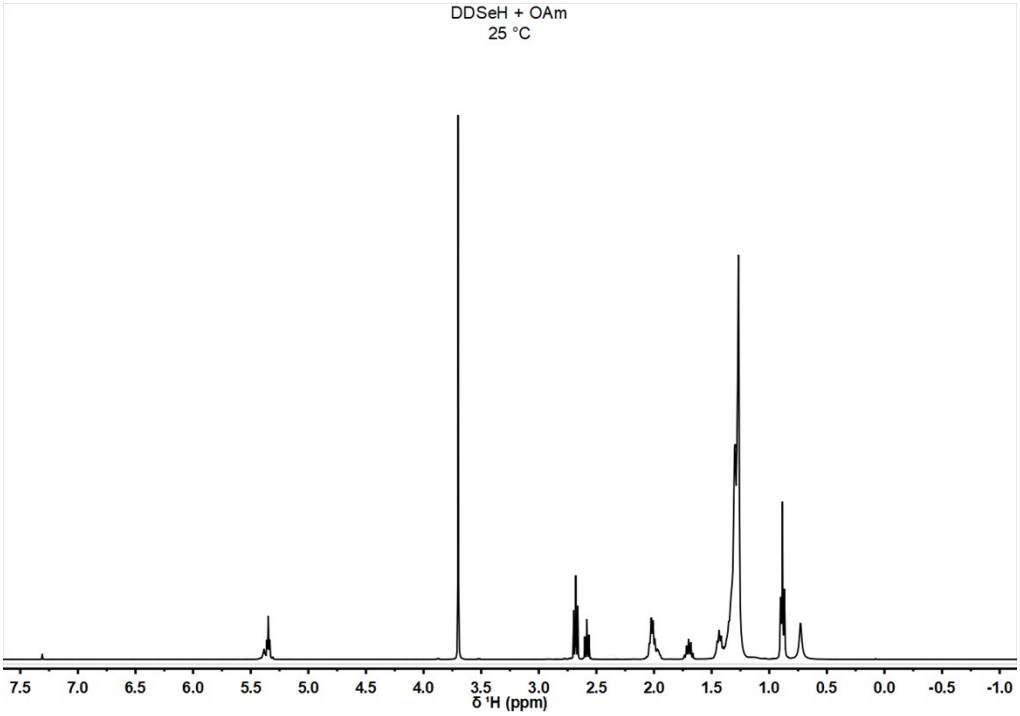

Figure S4: ^{1}H NMR of DDSeH and oleylamine at 25 °C in $CDCl_3$. Dioxane internal standard at δ = 3.71 ppm

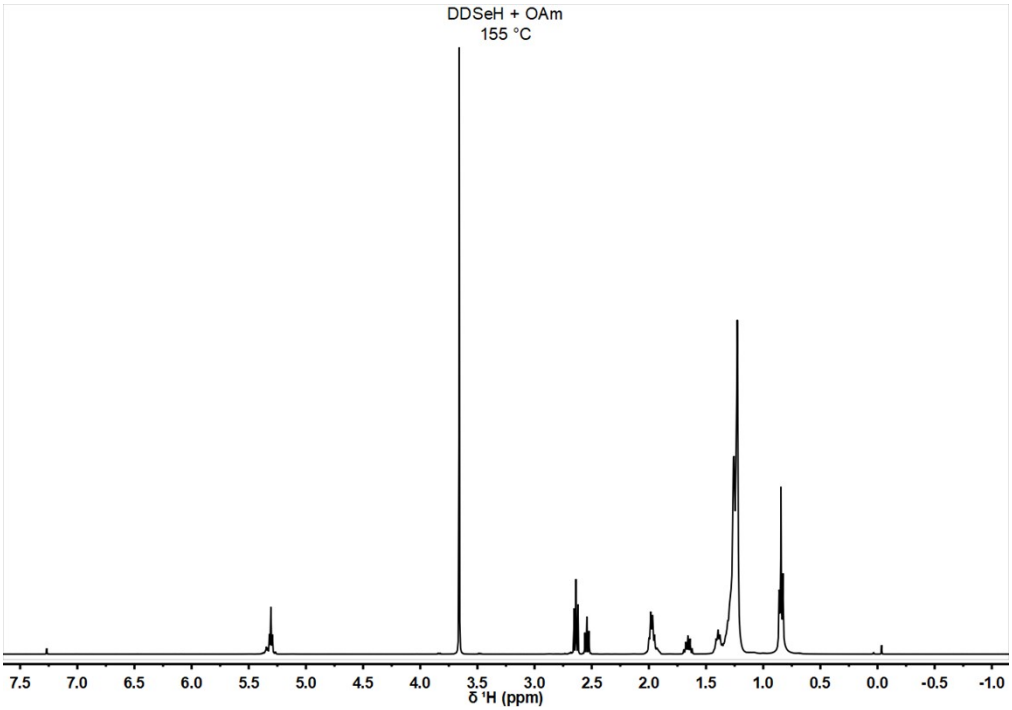

Figure S5: ^{1}H NMR of DDSeH and oleylamine at 155 °C in $CDCl_3$. Dioxane internal standard at δ = 3.71 ppm

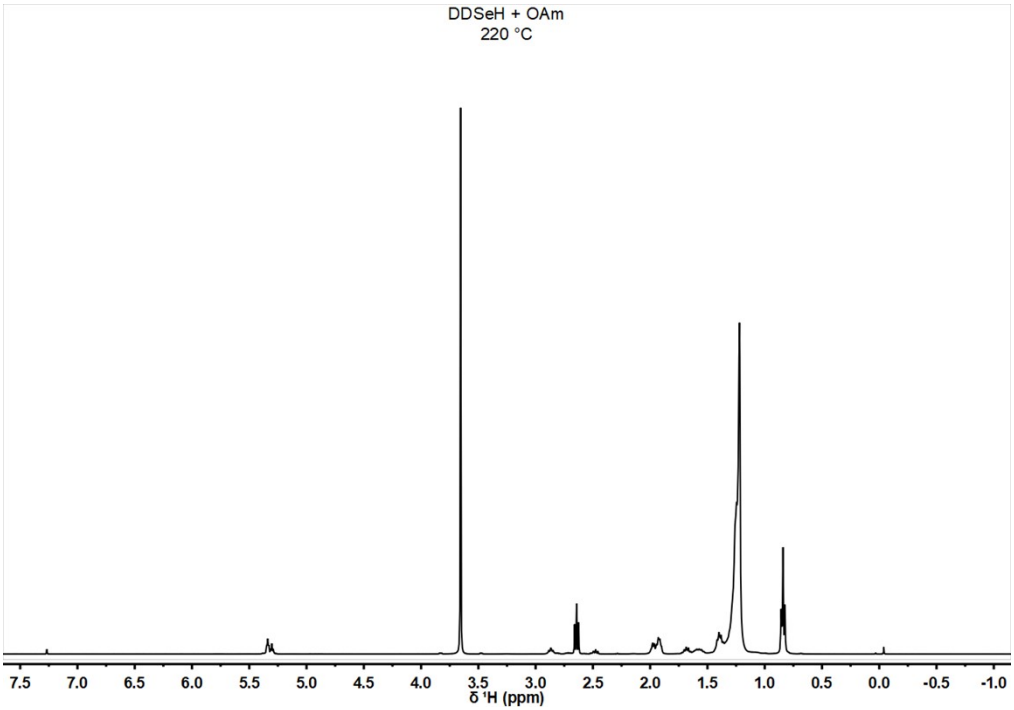

Figure S6: ^{1}H NMR of DDSeH and oleylamine at 220 °C in $CDCl_3$. Dioxane internal standard at δ = 3.71 ppm

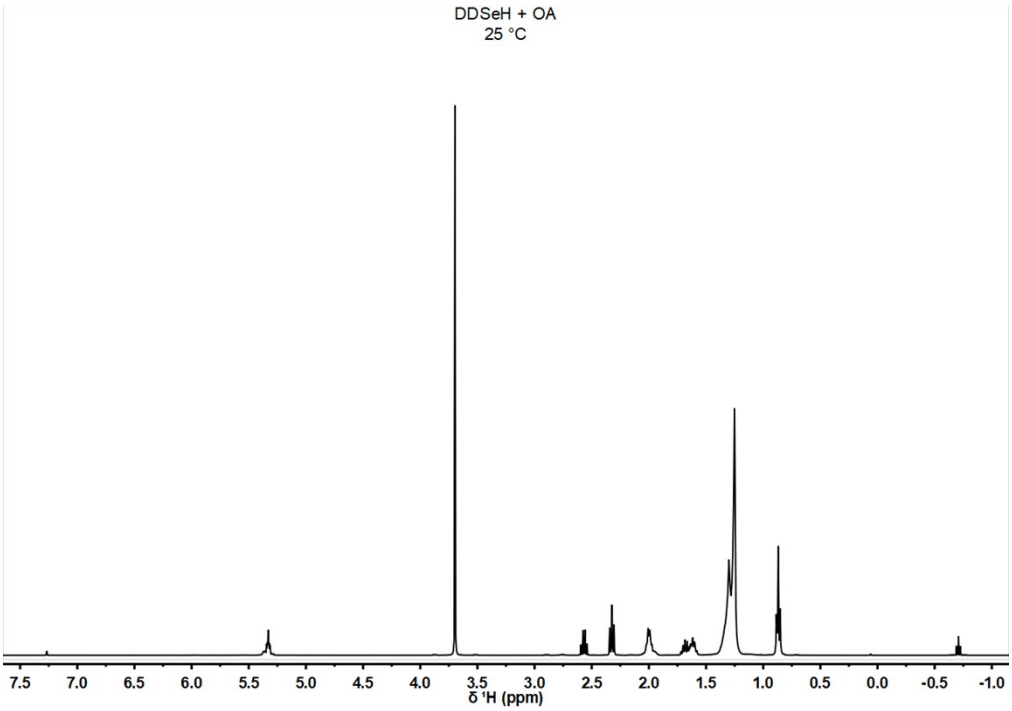

Figure S7: ^{1}H NMR of DDSeH and oleic acid at 25 °C in $CDCl_3$. Dioxane internal standard at δ = 3.71 ppm

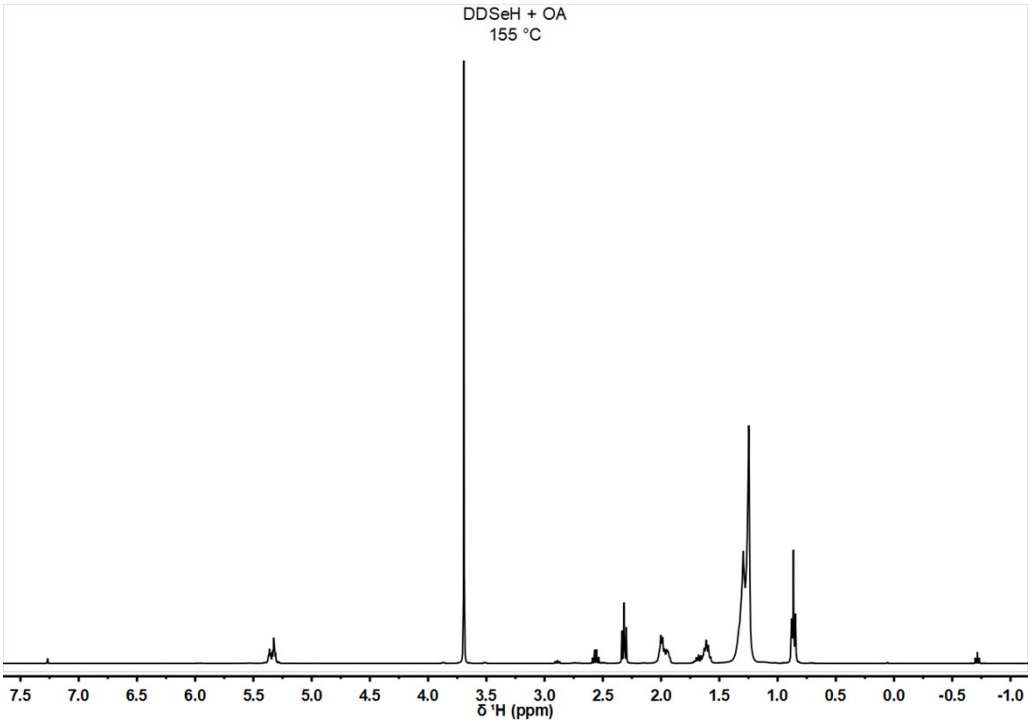

Figure S8: ^{1}H NMR of DDSeH and oleic acid at 155 °C in $CDCl_3$. Dioxane internal standard at δ = 3.71 ppm

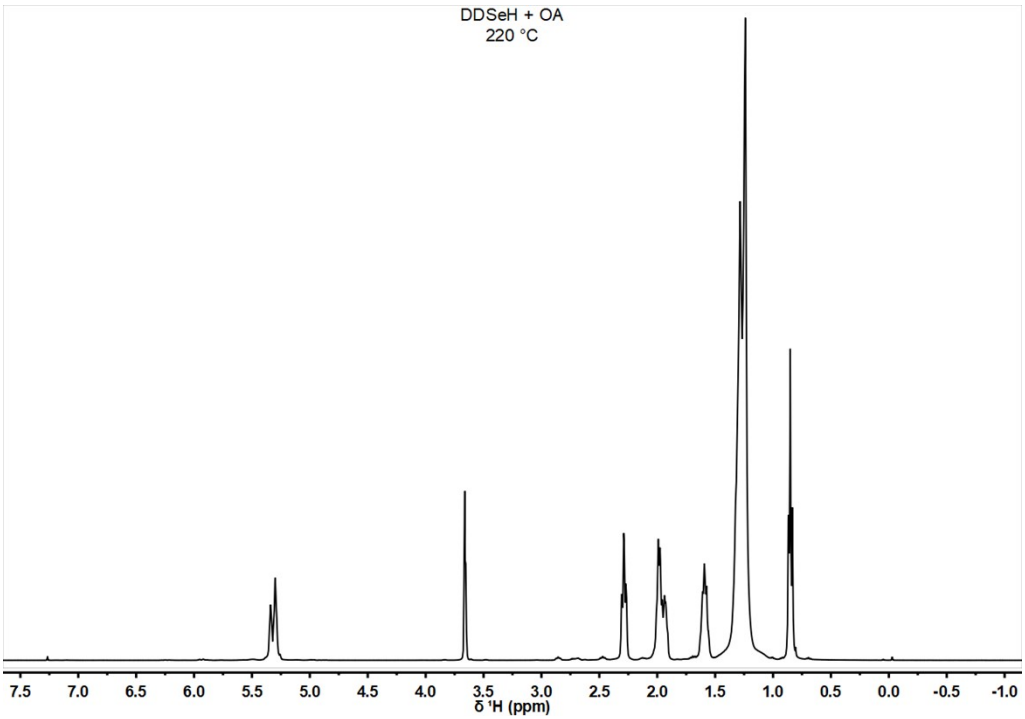

Figure S9: ^{1}H NMR of DDSeH and oleic acid at 220 °C in $CDCl_3$. Dioxane internal standard at δ = 3.71 ppm

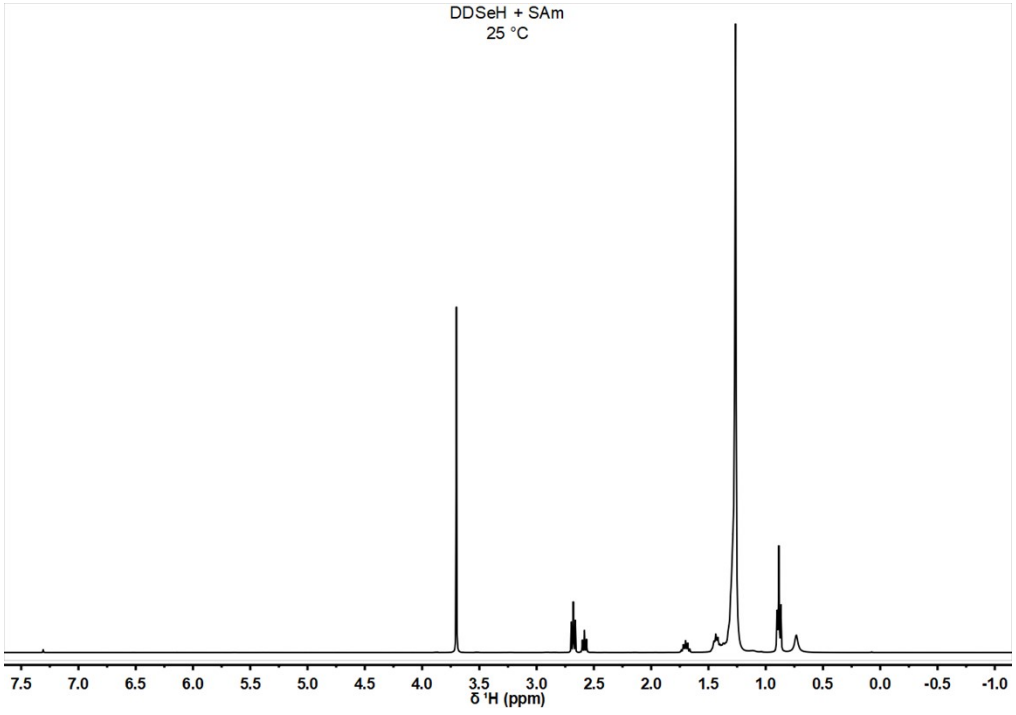

Figure S10: ^{1}H NMR of DDSeH and stearylamine at 25 °C in $CDCl_3$. Dioxane internal standard at δ = 3.71 ppm

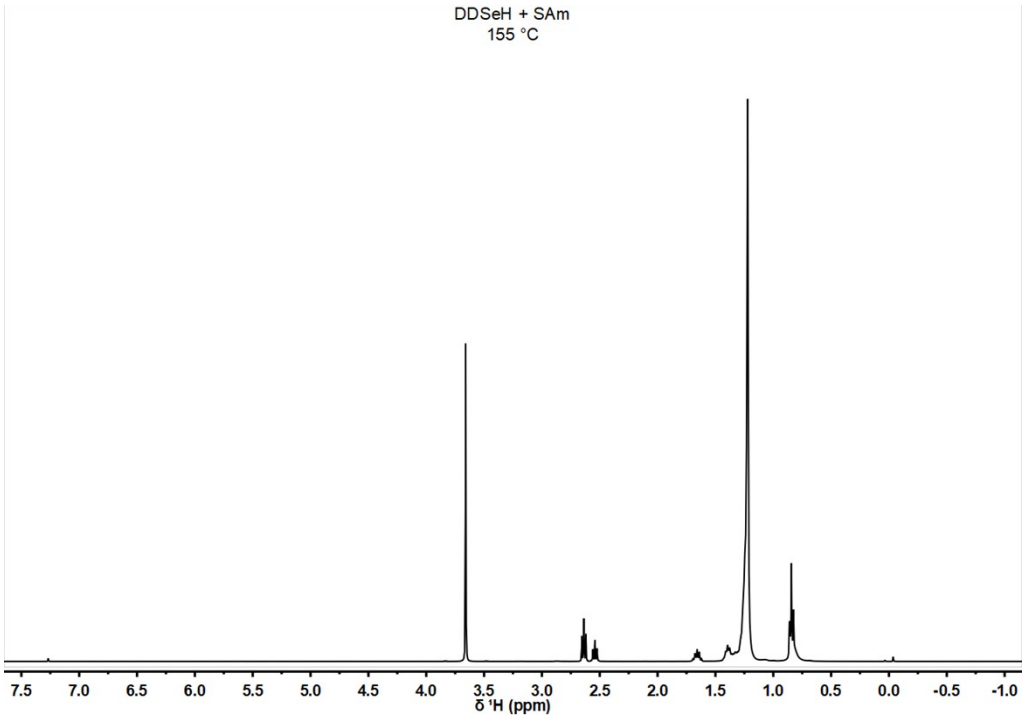

Figure S11: ^{1}H NMR of DDSeH and stearylamine at 155 °C in $CDCl_3$. Dioxane internal standard at δ = 3.71 ppm

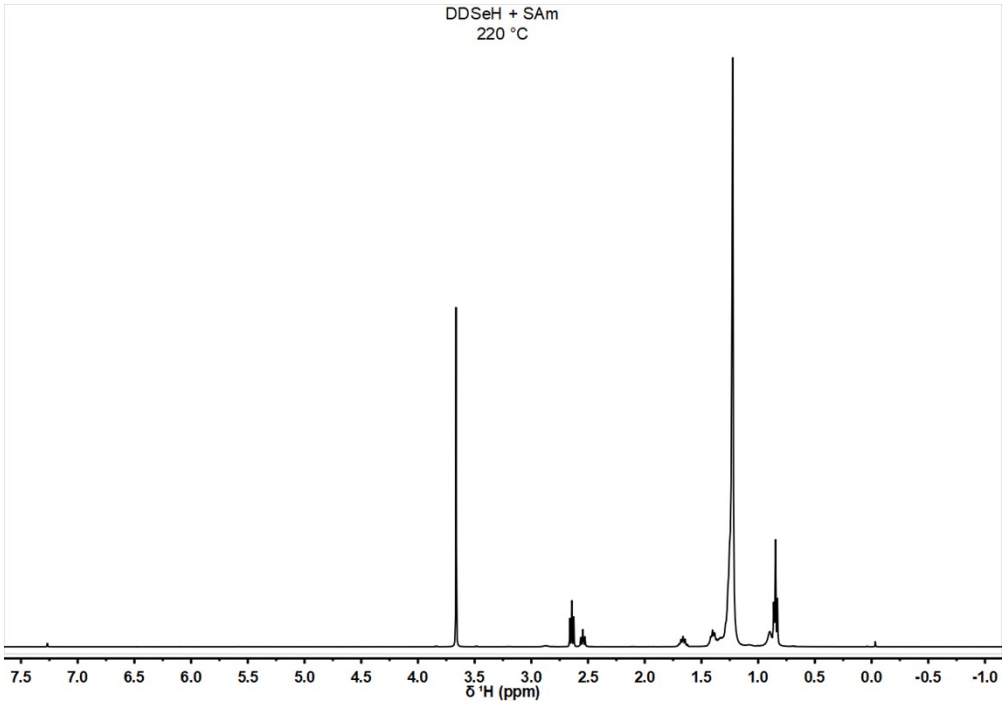

Figure S12: ^{1}H NMR of DDSeH and stearylamine at 220 °C in $CDCl_3$. Dioxane internal standard at δ = 3.71 ppm

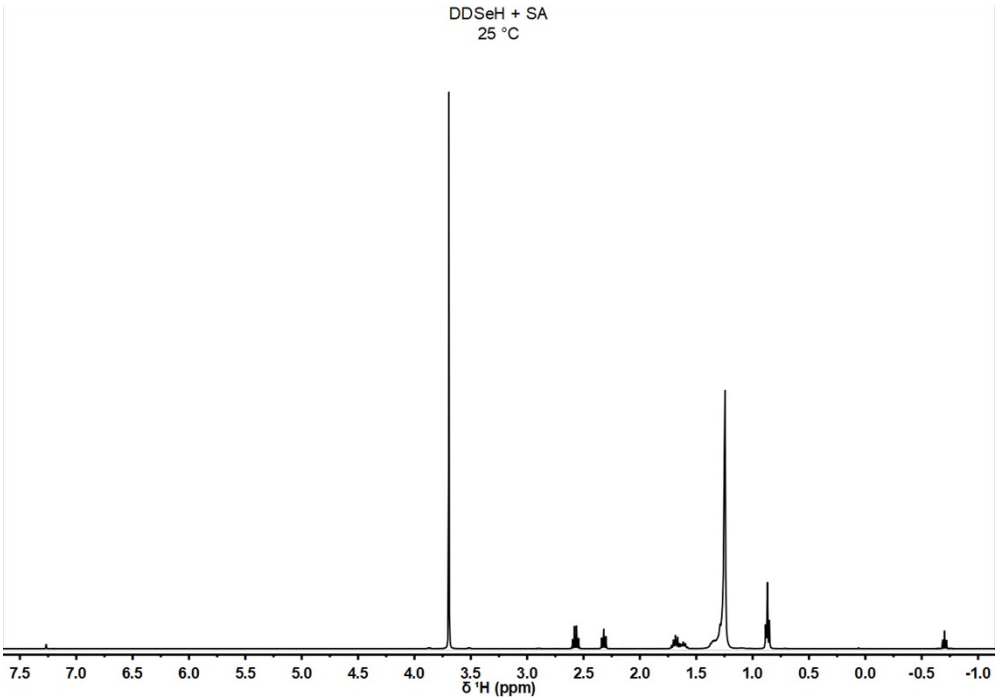

Figure S13: ^{1}H NMR of DDSeH and stearic acid at 25 °C in $CDCl_3$. Dioxane internal standard at δ = 3.71 ppm

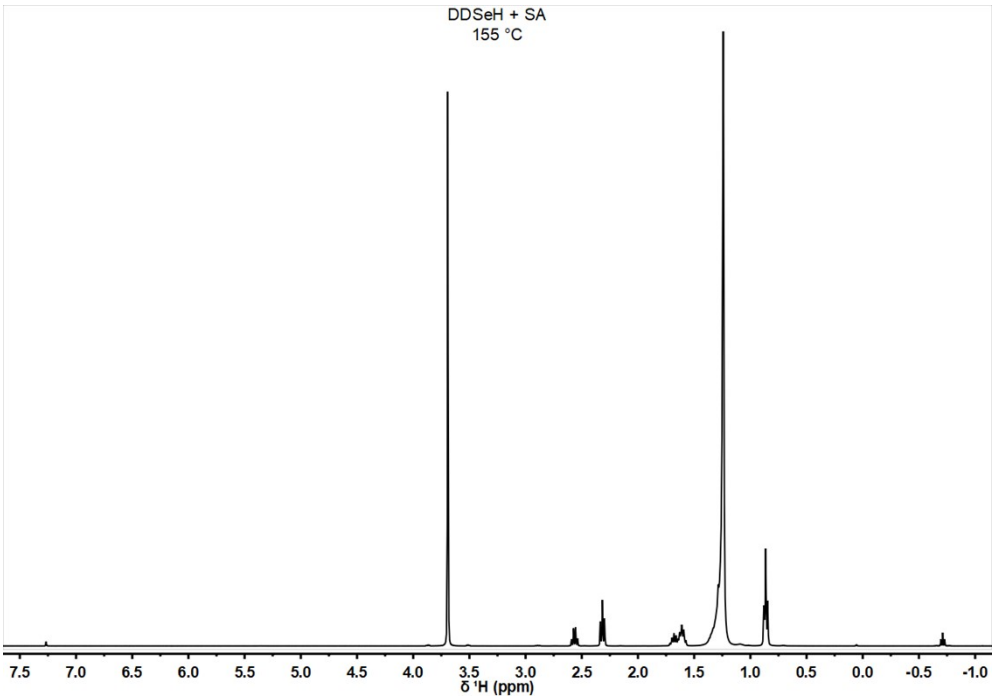

Figure S14: ^{1}H NMR of DDSeH and stearic acid at 155 °C in $CDCl_3$. Dioxane internal standard at δ = 3.71 ppm

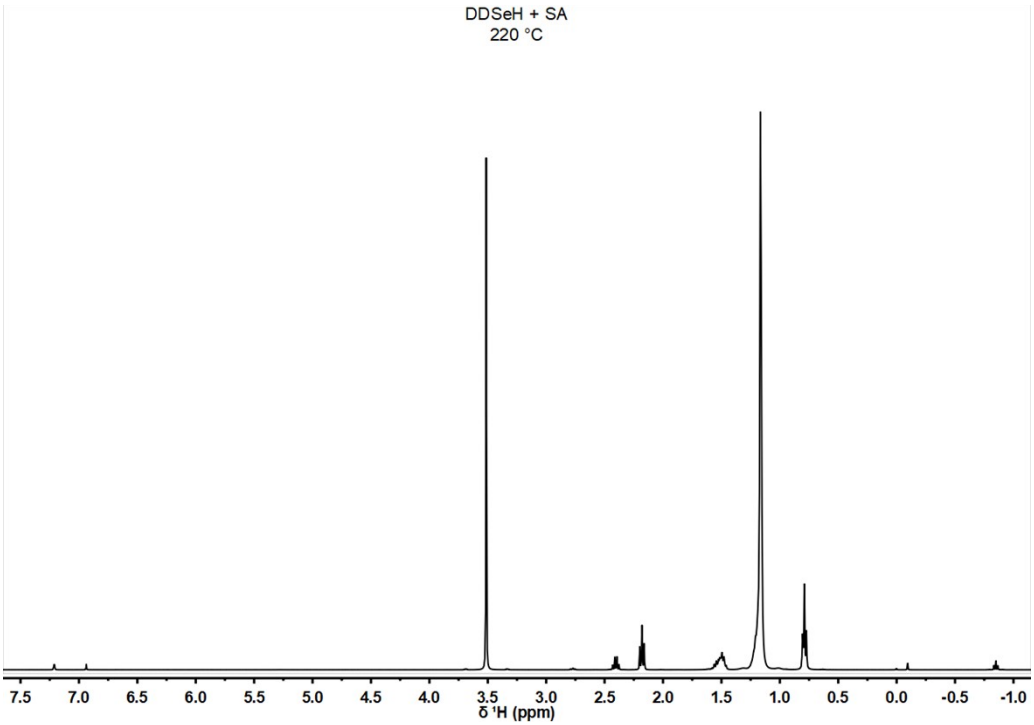

Figure S15: ^{1}H NMR of DDSeH and stearic acid at 220 °C in C_6D_6. Dioxane internal standard at δ = 3.71 ppm

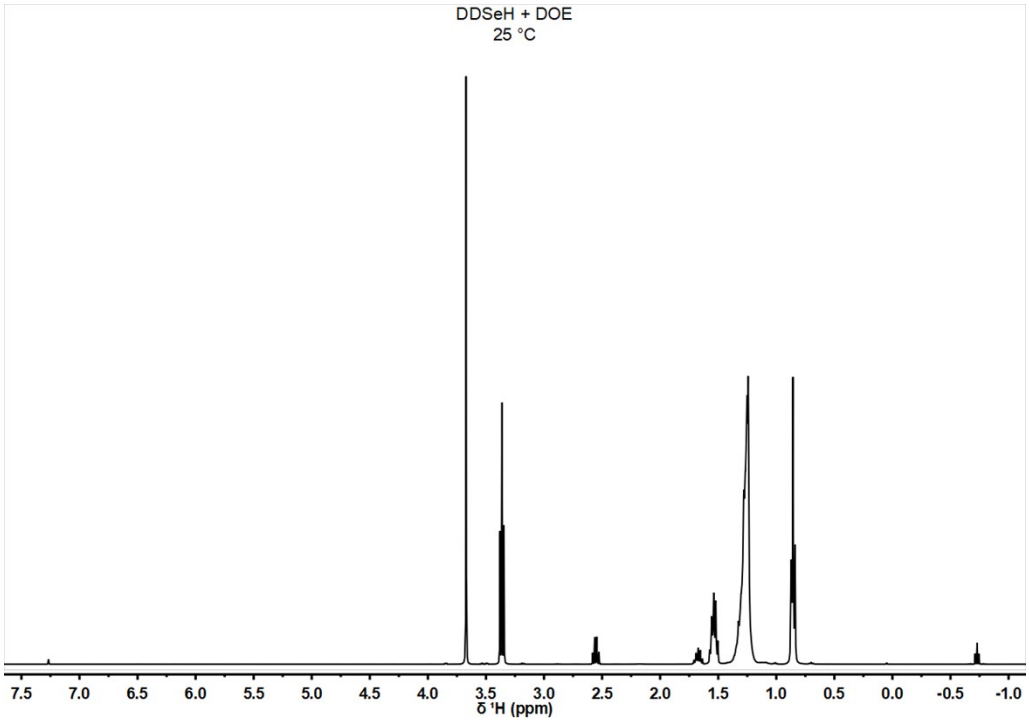

Figure S16: ^{1}H NMR of DDSeH and DOE at 25 °C in $CDCl_3$. Dioxane internal standard at δ = 3.71 ppm

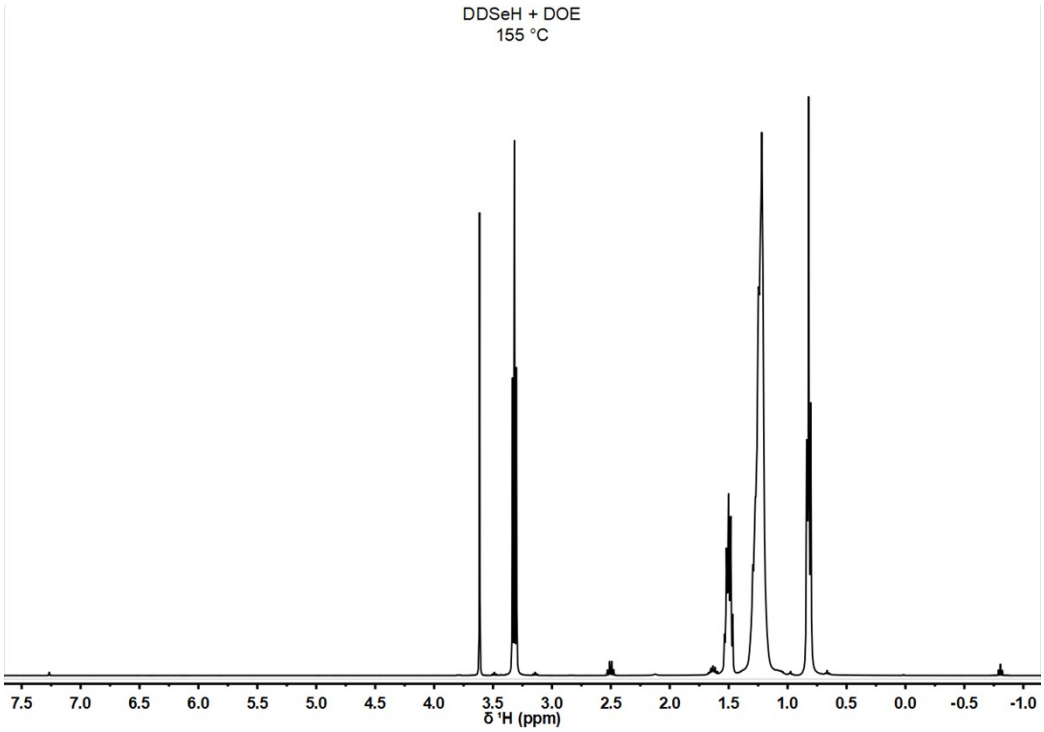

Figure S17: ^{1}H NMR of DDSeH and DOE at 155 °C in $CDCl_3$. Dioxane internal standard at δ = 3.71 ppm

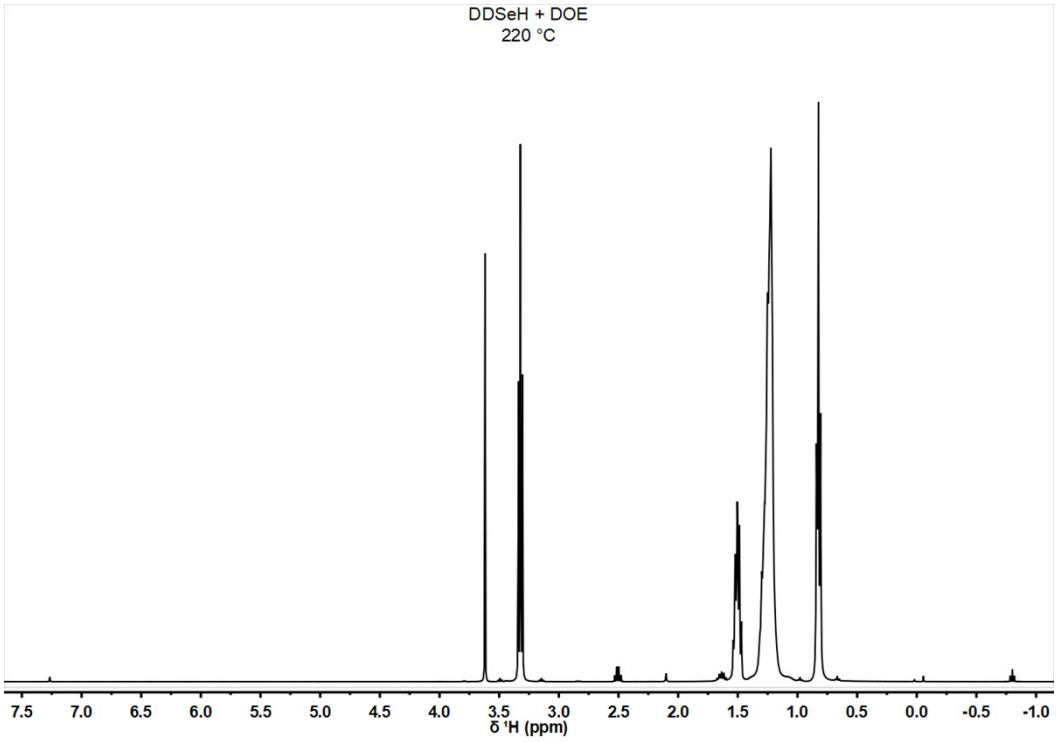

Figure S18: ^{1}H NMR of DDSeH and DOE at 220 °C in $CDCl_3$. Dioxane internal standard at δ = 3.71 ppm

^{77}H NMR of all DDSeH and ligand combinations tested at 25 °C

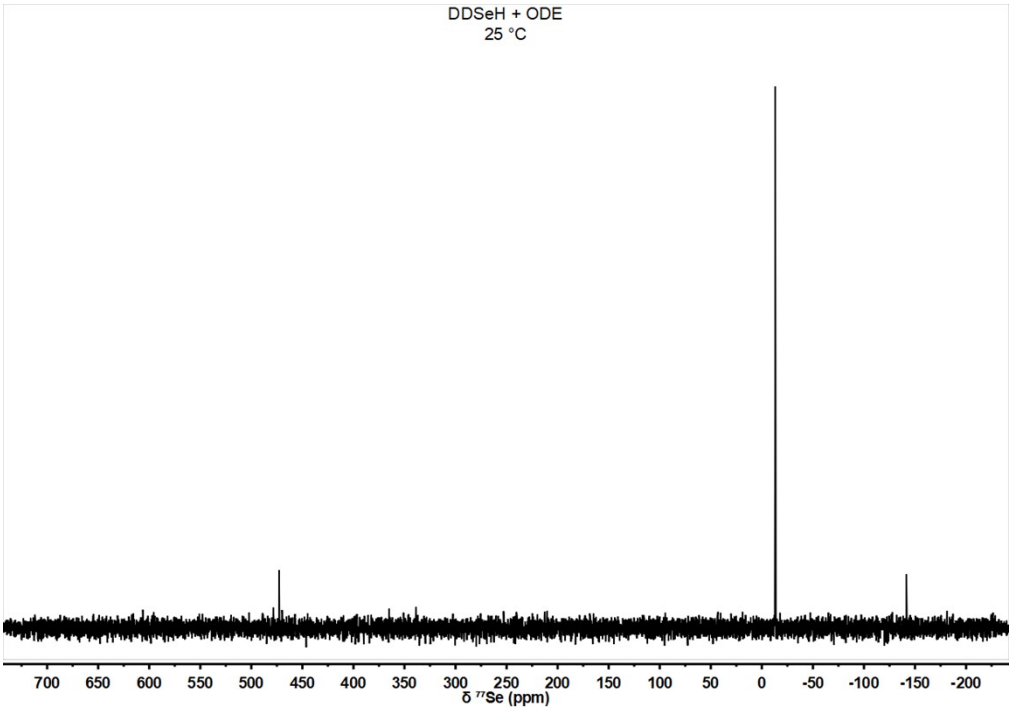

Figure S19: ^{77}Se NMR of DDSeH and ODE at 25 °C in $CDCl_3$.

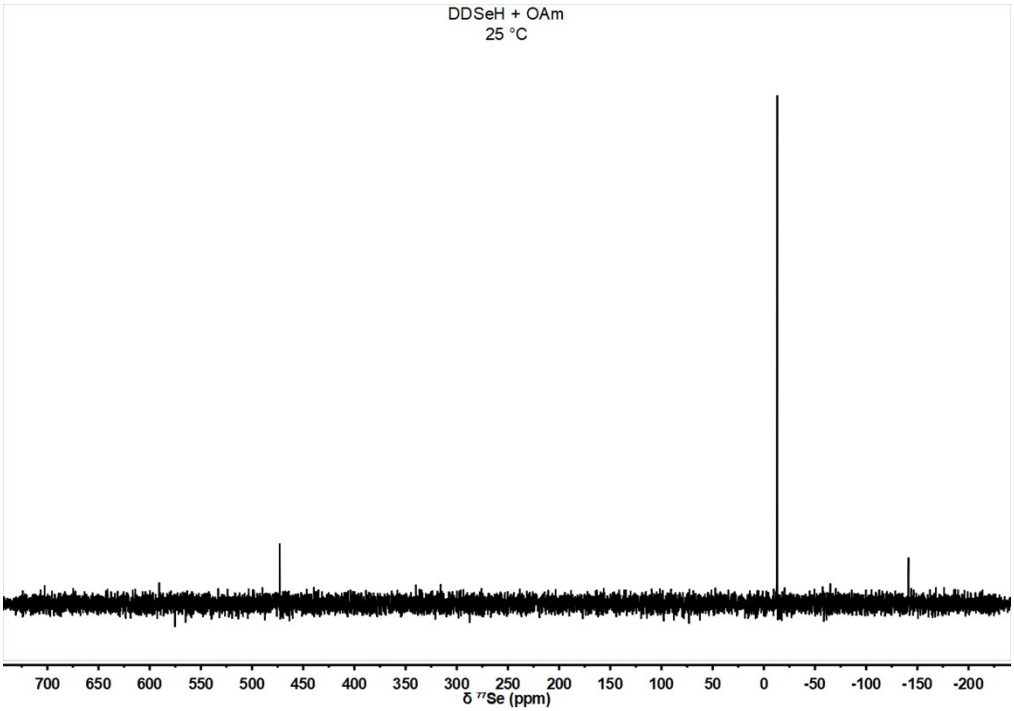

Figure S20: ^{77}Se NMR of DDSeH and ODE at 25 °C in $CDCl_3$.

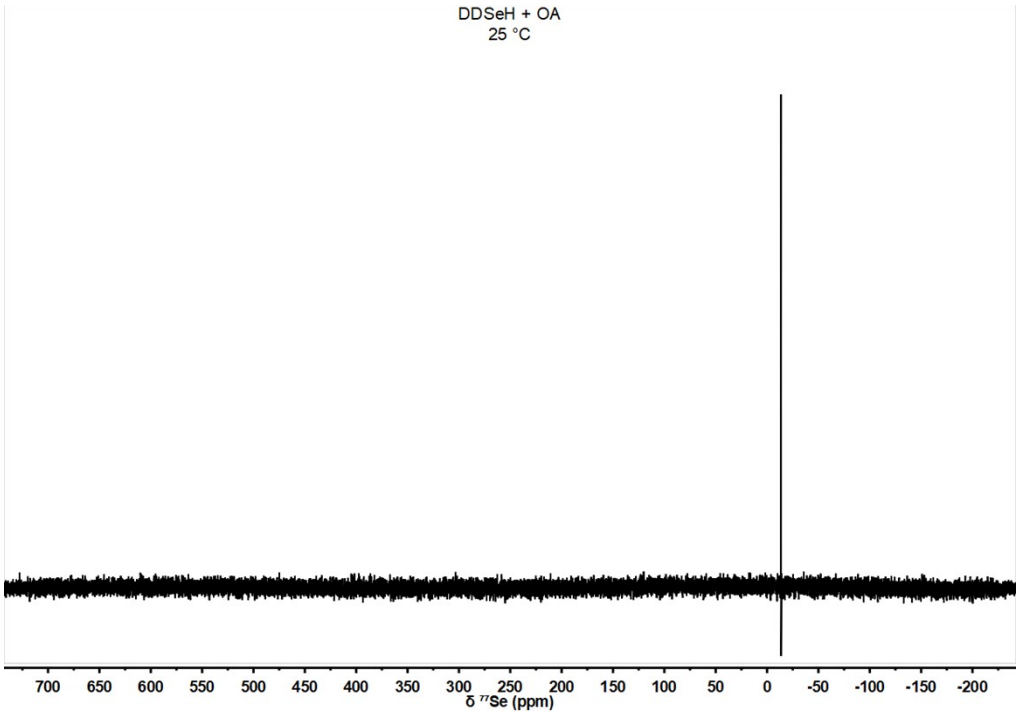

Figure S21: ^{77}Se NMR of DDSeH and oleic acid 25 °C in $CDCl_3$.

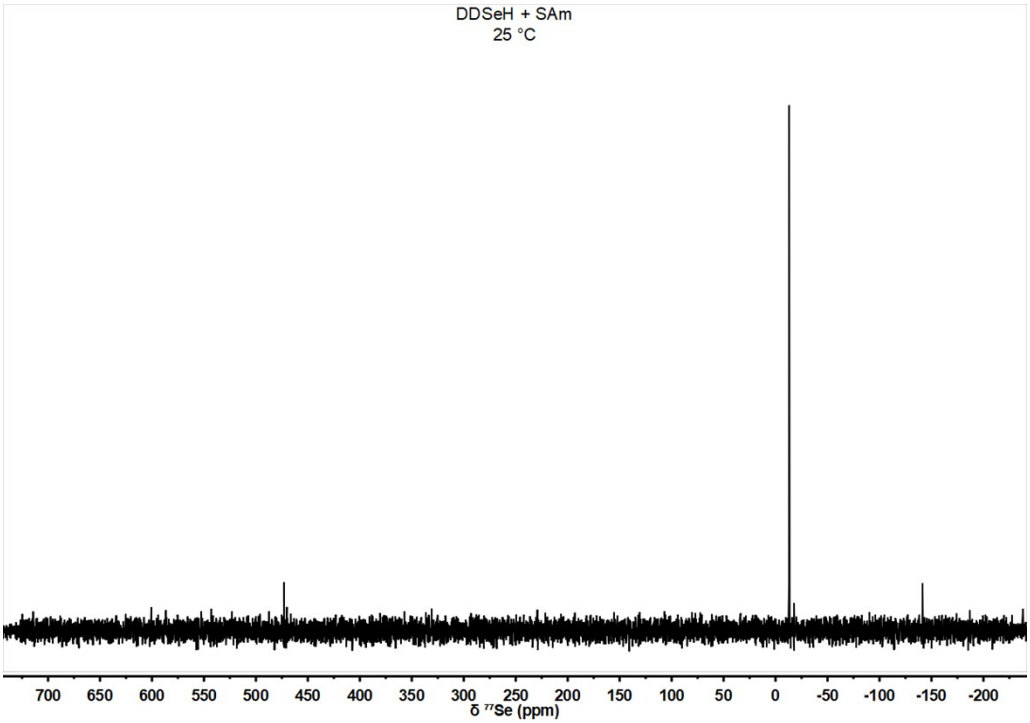

Figure S22: ^{77}Se NMR of DDSeH and stearylamine at 25 °C in $CDCl_3$.

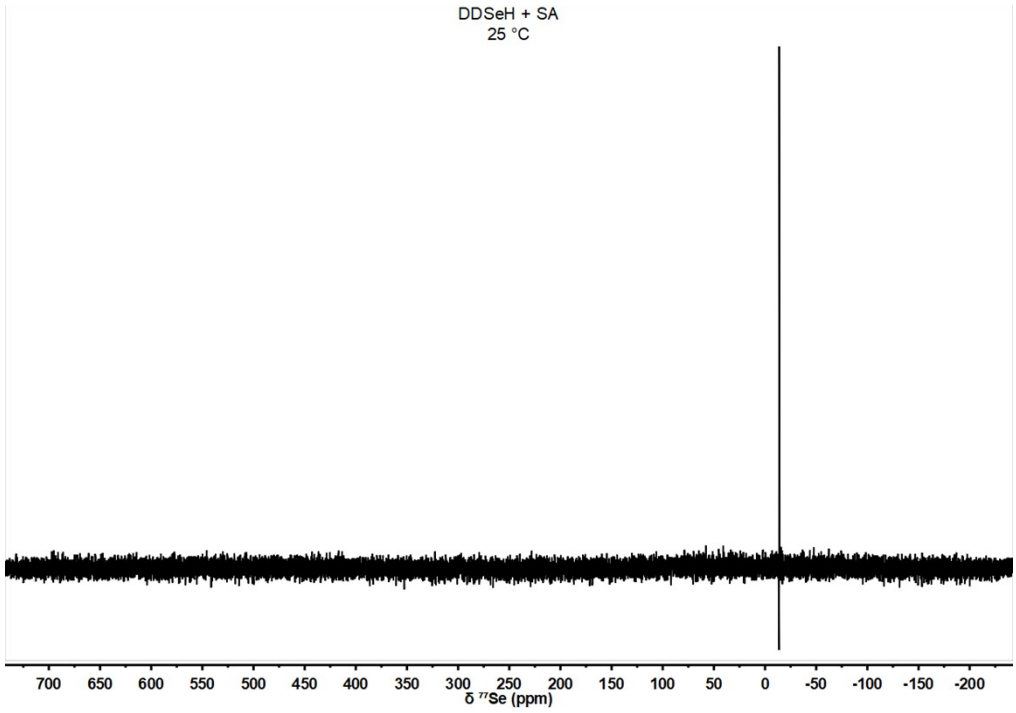

Figure S23: ^{77}Se NMR of DDSeH and stearic acid at 25 °C in $CDCl_3$.

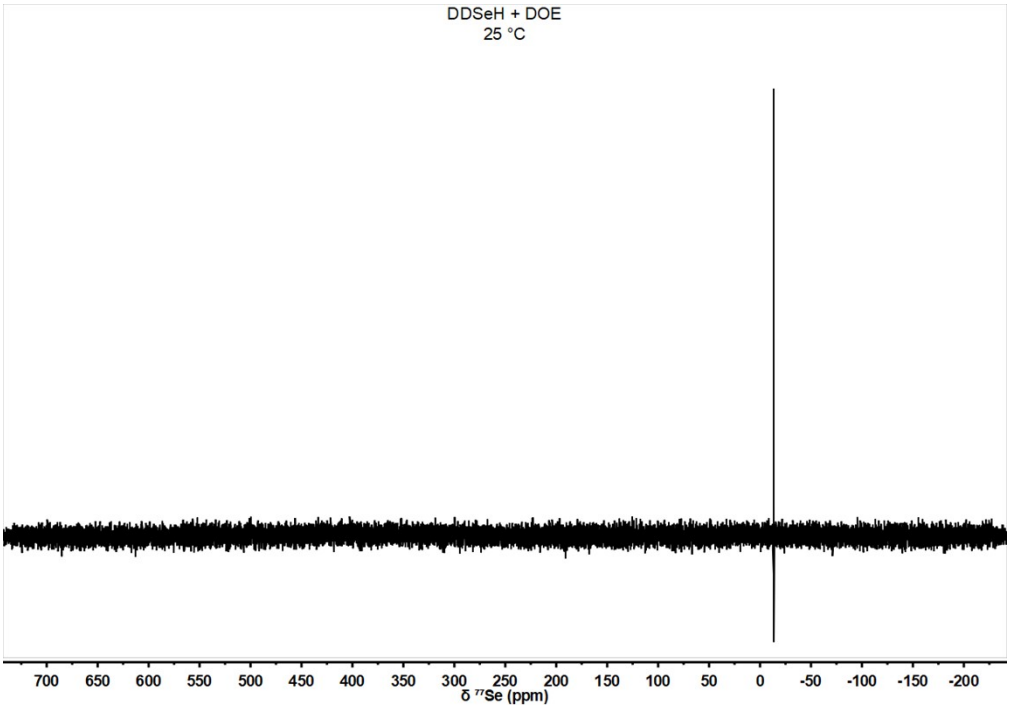

Figure S24: ^{77}Se NMR of DDSeH and DOE at 25 °C in $CDCl_3$.

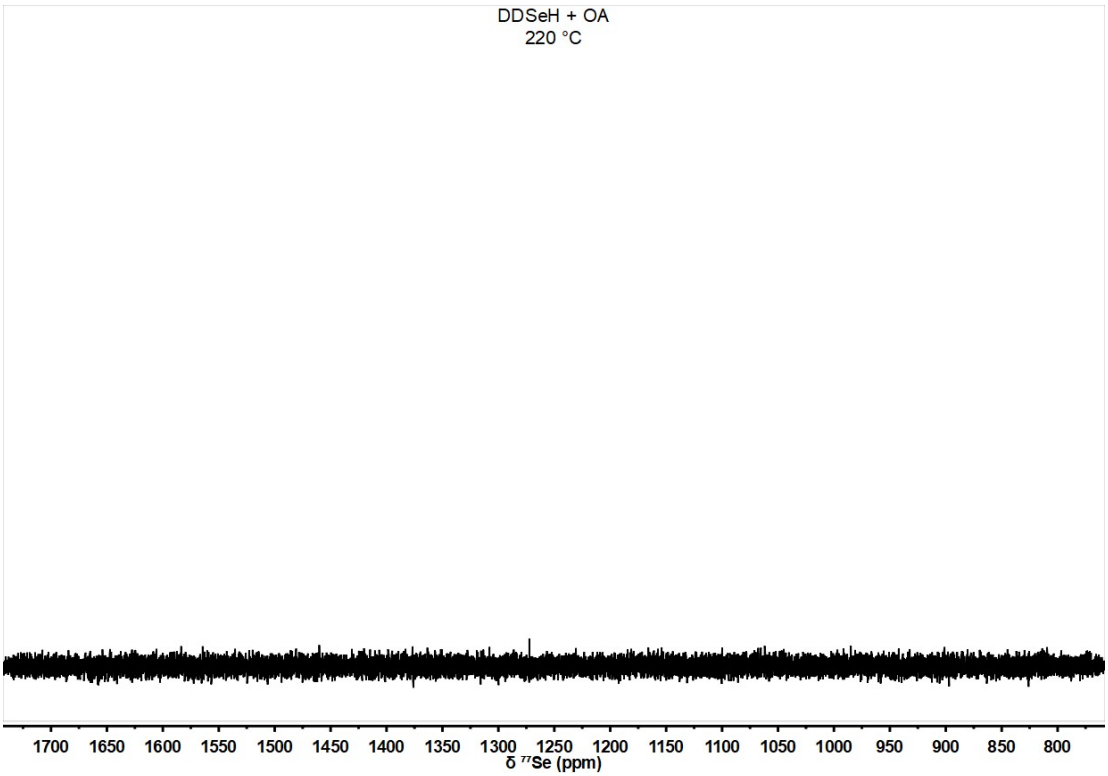

Figure S25: Upfield scan ^{77}Se NMR of DDSeH and oleic acid at 220 °C in $CDCl_3$

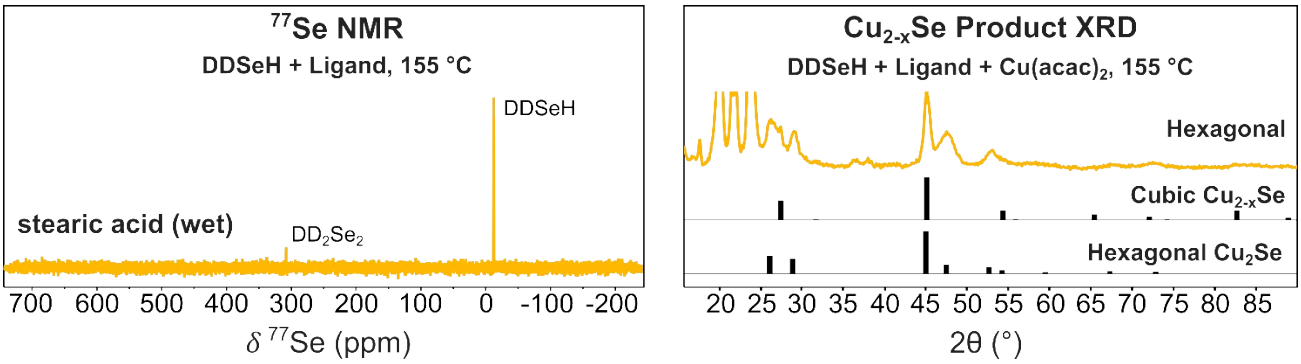

Figure S26: ^{77}Se NMR and pXRD experiments illustrating that failure to properly dry/degas stearic acid produces the hexagonal phase of $Cu_{2-x}Se$ nanocrystals.

pXRD Analysis of Lead Acetate Paper

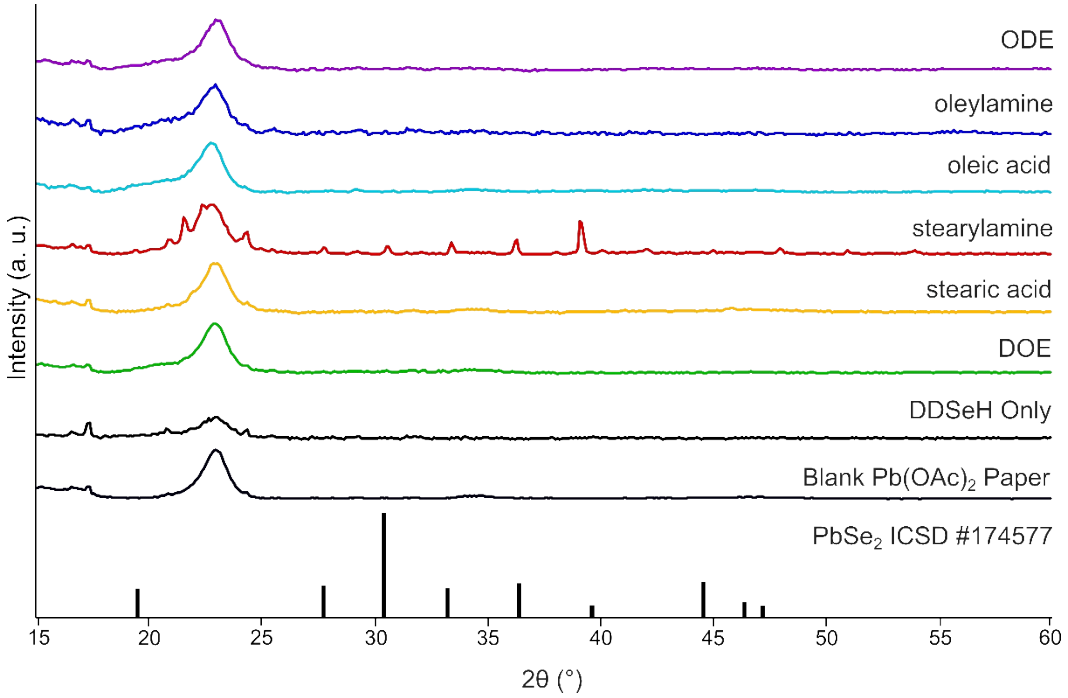

Figure S27: pXRD of lead acetate paper placed in reaction headspaces for studies at 155 °C. The formation of PbSe is indicative of H_2Se gas evolution.

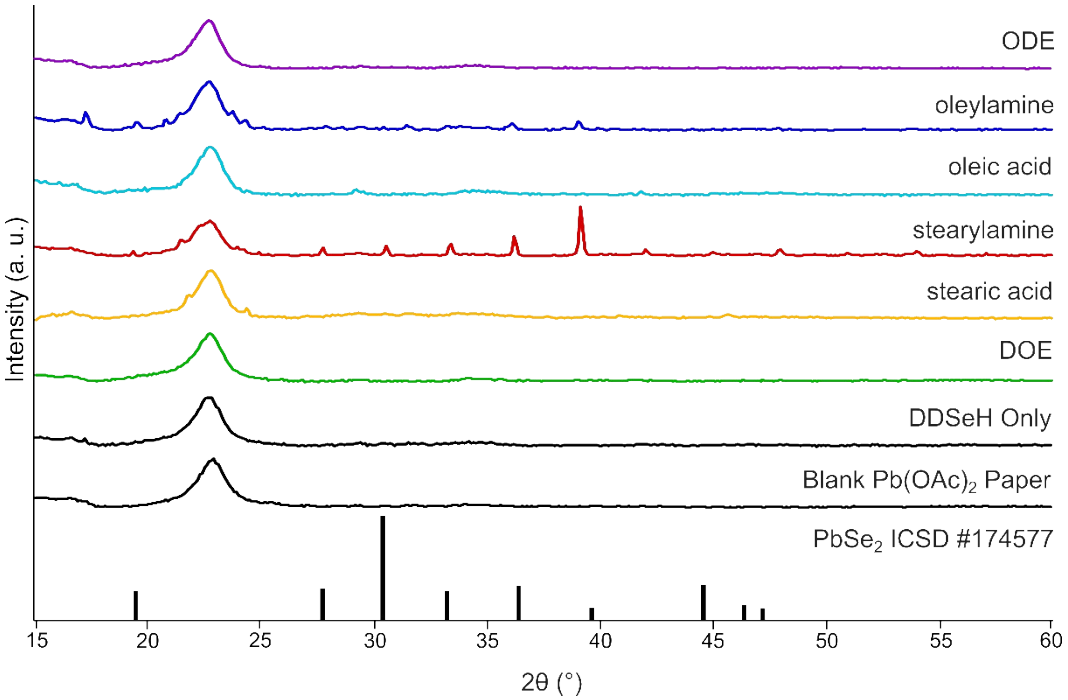

Figure S28: pXRD of lead acetate paper placed in reaction headspaces for studies at 220°C. The formation of PbSe is indicative of H_2Se gas evolution.

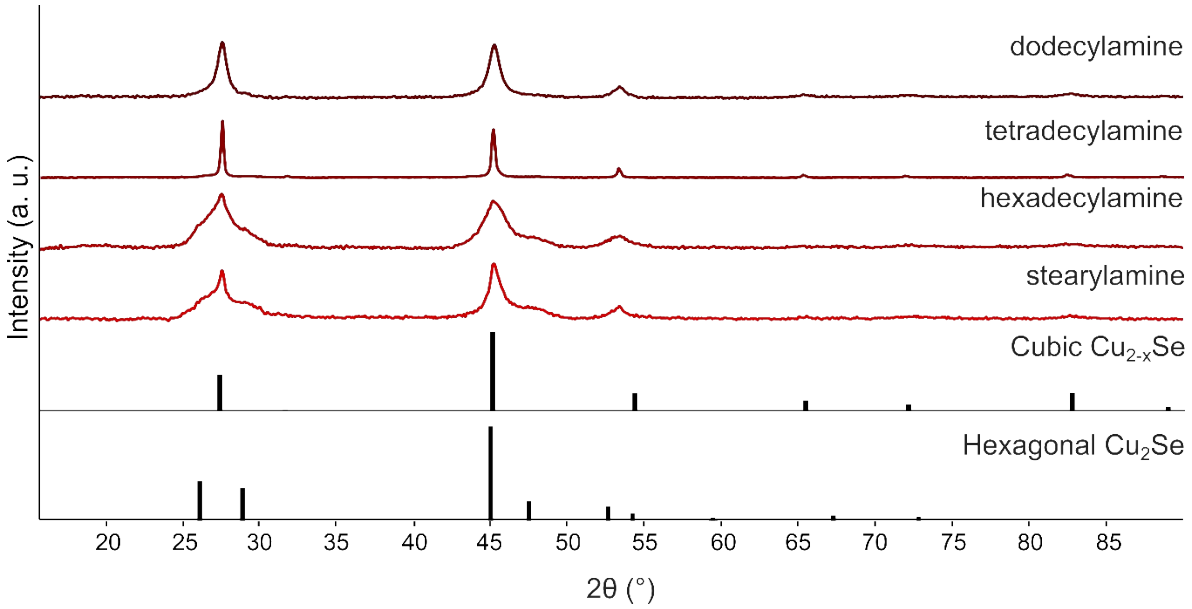

Figure S29: pXRD of copper selenide products from syntheses prepared in the presence of dodecylamine, tetradecylamine, hexadecylamine and stearylamine. Calculated patterns for cubic berzelianite $Cu_{2-x}Se$ and hexagonal $Cu_{2-x}Se$ used for Rietveld refinements of percent composition reported in Figure 3. All refinements have $X^2 < 3$.

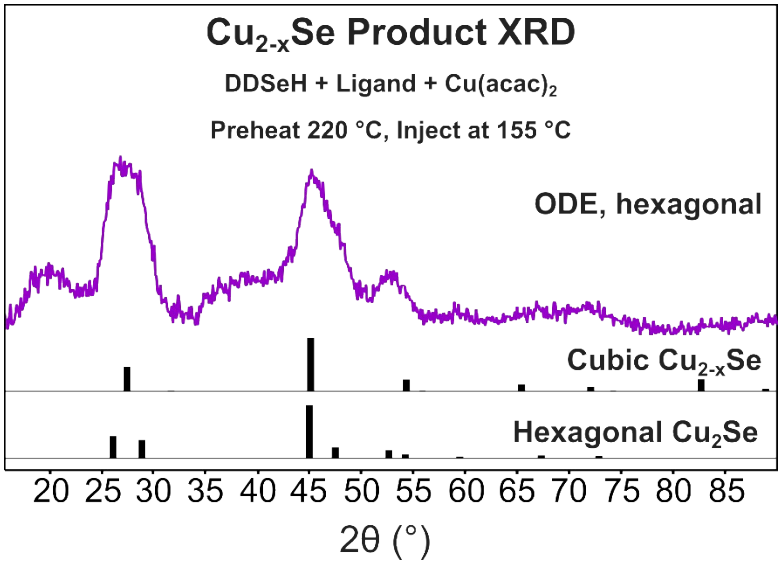

Figure S30: pXRD of copper selenide products from synthesis using ODE. DDSeH and ODE were preheated for 1 hr at 220 °C then cooled to 155 °C before Cu precursor injection.