

## Electronic Supplementary Information

### Polysalt Ligands Achieve Higher Quantum Yield and Improved Colloidal Stability for CsPbBr<sub>3</sub> Quantum Dots

Sisi Wang,<sup>1</sup> Liang Du,<sup>1</sup> Selin Donmez,<sup>1</sup> Yan Xin<sup>®</sup> and Hedi Mattoussi

<sup>1</sup> Florida State University, Department of Chemistry and Biochemistry, 95 Chieftan Way, Tallahassee, FL 32306

<sup>®</sup> Florida State University, National High Magnetic Field Laboratory, 1800 E. Paul Dirac Drive, Tallahassee, Florida, 32310

#### Materials

Cs<sub>2</sub>CO<sub>3</sub> (99.9%), PbBr<sub>2</sub> (99.99%), octadecene (ODE, 90%), oleic acid (OA, 90%), oleylamine (OLA, 70%), octylamine (OCA, 99.9%), octadecylamine (ODA, 99%), Poly(isobutylene-*alt*-maleic anhydride) (PIMA) (average MW: ~6000 Da), N,N-dimethylamino propylamine, 1-(3-Aminopropyl) imidazole, bromoethane along with most of the chemicals used were purchased from Sigma Aldrich (St Louis, MO). Solvents used in this study were also acquired from Sigma Aldrich (St Louis, MO). Deuterated solvents used for NMR experiments were purchased from Cambridge Isotope Laboratories (Andover, MA). The chemicals and solvents were used as purchased without further purification. Synthesis of the various compounds was carried out under N<sub>2</sub> passed through an O<sub>2</sub> scrubbing tower, unless otherwise stated. Air sensitive materials were handled in an Mbraun Labmaster glovebox, and standard Schlenk techniques were used when handling air-sensitive materials. Column purification chromatography applied to certain compounds was performed using silica gel (60 Å, 230–400 mesh), purchased from Bodman Industries (Aston, PA).

#### Characterization

The optical absorption data were collected using a UV-Vis absorption spectrophotometer (UV 2450 model, Shimadzu, Columbia, MD). The steady-state fluorescence spectra were collected using a Fluorolog-3 spectrofluorometer (HORIBA Jobin Yvon, Edison, NJ) equipped with a fast TBX PMT detector. The time-resolved fluorescence decay profiles were acquired and analyzed using a Time Correlation Single Photon Counting (TCSPC) system integrated into the Fluorolog-3 spectrofluorometer. Sample excitation was carried out using a pulsed laser source NanoLED-

440LH (100 ps, FWHM), which generates a signal at 440 nm with a repetition rate of 1 MHz, while detection was collected on the TBX detector. The system can resolve lifetimes down to 40-50 ps. The fluorescence decay profiles were limited to a narrow spectral window centered at the materials emission, and best fitted to a bi-exponential function:

$$I(t) = A_1 e^{-\frac{t}{\tau_1}} + A_2 e^{-\frac{t}{\tau_2}} \quad (1)$$

where  $t$  is time and  $A_i$  is a weighting parameter associated with each decay time,  $\tau_i$ . An average amplitude-weighted lifetime,  $\tau$ , was extracted from the fitting:

$$\tau = \frac{\sum A_i \tau_i^2}{\sum A_i \tau_i} \quad (2)$$

The PL quantum yield (QY) measurements were carried out using a Quantaurus absolute PL quantum yield spectrometer (C11347) from Hamamatsu (Bridgewater, NJ). The setup acquires an integrated signal over the photoemission spectrum, which is then normalized with respect to the excitation profile, as previously detailed;<sup>1</sup> the excitation wavelength was at 400 nm. The QY values of the NC dispersions were extracted from the intensities of the signals emitted,  $I(\text{Em})$ , and absorbed,  $I(\text{Abs})$ , by the sample using the equation:

$$\Phi = \frac{I(\text{Em})}{I(\text{Abs})} = \frac{\int_{\lambda_3}^{\lambda_4} \left(\frac{\lambda}{hc}\right) \times \{I_S^{\text{T-Corr}}(\lambda) - I_R^{\text{T-Corr}}(\lambda)\} d\lambda}{\int_{\lambda_1}^{\lambda_2} \left(\frac{\lambda}{hc}\right) \times \{I_R^{\text{T-Corr}}(\lambda) - I_S^{\text{T-Corr}}(\lambda)\} d\lambda} \quad (3)$$

Where  $h$  and  $c$  respectively designate the Planck constant and speed of light. The wavelength intervals  $[\lambda_1, \lambda_2]$  and  $[\lambda_3, \lambda_4]$  designate the range limits for the excitation and emission spectra, respectively.  $I_R^{\text{T-cor}}(\lambda) = I_R(\lambda)/T(\lambda)$  and  $I_S^{\text{T-cor}}(\lambda) = I_S(\lambda)/T(\lambda)$  are respectively the corrected intensities of the excitation and sample emission profiles, accounting for the transmission of the sample/reference,  $T(\lambda)$ .  $I_R(\lambda)$  and  $I_S(\lambda)$  refer to the intensity of the excitation source and sample emission (which also includes a small contribution from the excitation signal).

The  $^1\text{H}$  NMR spectra were recorded using a 600 MHz spectrometer (Bruker SpectroSpin, Billerica, MA). The dynamic light scattering measurements were carried out using an ALV/CGS-3 Compact Goniometer System (ALV-GmbH, Langen, Germany), equipped with a HeNe laser (illumination at 632.8 nm), ALV photon correlator and an avalanche photodiode for signal detection. The intensity autocorrelation profile at a given scattering angle  $\theta$ ,  $g^{(2)}(\theta, \tau)$  vs  $\log(\tau)$ , is built using the average of 3 acquisitions of 10 seconds each. Histograms of the intensity vs hydrodynamic size (Int. vs  $R_H$ ) profiles were extracted from  $g^{(2)}(\theta, \tau)$  using the Laplace transform.<sup>2</sup> The Stokes-Einstein relation is used to convert the diffusion coefficient,  $D$ , to  $R_H$  in the histograms:  $D = k_B T / 6\pi\eta R_H$ , where  $k_B$  and  $\eta$  respectively designate the Boltzmann constant and viscosity of the solvent used.

The powder X-ray diffraction patterns (PXRD) measured for the PQD samples were collected on a Rigaku MiniFlex 6G, equipped with a D/TeX Ultra 2 silicon strip detector. The diffraction patterns

were recorded over a  $2\theta$  range of  $10^\circ$ – $60^\circ$  using Cu-K $\alpha$  radiation ( $k = 1.5406 \text{ \AA}$ ) that was operated at 15 mA and 40 kV using Bragg–Brentano geometry. A step size of  $0.05^\circ$  and a scan rate of  $5^\circ/\text{min}$  was used for the measurements. The OLA/OA–PQDs samples were prepared by drop casting a concentrated dispersion of nanocrystals onto a glass slide and letting it dry. A polysalt-stabilized PQD sample was typically prepared by first precipitating the NCs using a non-solvent, and then the resulting pellet was dried to form a solid.

The TEM images of the various PQD samples were acquired using a JEOL JEM-ARM200cF (a cold field emission probe Cs-Corrected Transmission Electron Microscope, Peabody, MA) operated at 200 kV. The high angle annular dark field scanning transmission electron microscopy (HAADF-STEM) images were collected with a JEOL HAADF detector using the following experimental conditions: probe size 0.78 nm, scan speed 32 ms per pixel, and camera length 8 cm, which corresponds to a probe convergence angle of 21 mrad and inner collection angle of 76 mrad. To prepare a TEM sample a drop of a dispersion of either OLA/OA-capped PQDs in hexane, or the polysalt-stabilized PQDs in ethanol is deposited onto a TEM grid (200 Cu mesh, Ted Pella, INC, CA) and letting it slowly dry under a mild vacuum.

## Synthesis of the Polysalt Ligands L1-L6

The ligands prepared and tested in this report present different numbers of salt groups along with the complementary numbers of solubilizing alkyl chains (octylamine, OCA, or octadecylamine, ODA), installed along the PIMA chain. We identify each ligand by referring to the molar percentage of salts and alkyl chains with respect to the monomer concentration of the starting PIMA. The description below details the synthesis of the precursor polymers followed by the polysalts, via conversion of tertiary amines and imidazoles to the corresponding ammonium and imidazolium bromide salts (AMB and IMB). Below we detail the synthesis of the polymers with the various stoichiometric molar combinations of AMB (or IMB) with ODA (or OCA).

**1) Synthesis of AM20%-PIMA-ODA80% and of IM20%-PIMA-ODA80%.** PIMA (1g, 6.64 mmol of monomer units) was first dissolved in 5 mL of DMF using a 100 mL round bottom (RB) flask equipped with a stirring bar, followed by the addition of 1.33 mmol of either N, N-dimethylaminopropylamine (AM), or 1-(3-Aminopropyl) imidazole (IM) pre-dissolved in 1 mL of DMF. The reaction mixture was heated to  $60^\circ\text{C}$  and left stirring for 2h. Then, octadecylamine (ODA, 1.44 g, 5.33 mmol) dissolved in 5 mL of  $\text{CHCl}_3$  was added. A slight heating was first applied to the ODA and chloroform mixture in order to obtain a clear solution. The reaction was then left stirring overnight at  $60^\circ\text{C}$ . The solvent was evaporated under vacuum and the compound was dissolved in 5 mL of  $\text{CHCl}_3$ . One round of column chromatography using pure  $\text{CHCl}_3$  as eluent was applied, the eluted fractions were combined, and the solvent was evaporated. The residue was further dried under vacuum, yielding the final product as a white powder; the overall reaction yield is  $\sim 55\%$ .

The reaction conditions used above yield polymers where 8 of the anhydride rings (i.e., 20% of the total number of monomers) along PIMA have been reacted to introduce AM or IM groups, while the other 80% of the rings were reacted with ODA, yielding compounds with the stoichiometry: AM20%-PIMA-ODA80% or IM20%-PIMA-ODA80%. Varying the relative molar fraction of N, N-dimethylaminopropylamine, or 1-(3-Aminopropyl) imidazole with respect to ODA during the reaction will allow the preparation of polymers with other stoichiometries.

**2) Synthesis of L1: AMB20%-PIMA-ODA80% and L4: IMB20%-PIMA-ODA80%.** Conversion of AM-PIMA-ODA to AMB-PIMA-ODA, or IM-PIMA-ODA to IMB-PIMA-ODA was carried out using similar protocols. Briefly, ~1.5 g of AM-PIMA-ODA (or IM-PIMA-ODA) obtained in the step 1 above, dissolved in anhydrous THF (10 mL), was loaded onto a pressure vessel, and then 5 mL of bromoethane was added. The reaction mixture was heated to 70 °C and left stirring overnight. The vessel was cooled to room temperature and the unreacted bromoethane was removed using a rotary evaporator. The product dissolved in small amount of CHCl<sub>3</sub> was washed using excess ethyl acetate (one round) and dried under vacuum, yielding the polysalt (AMB-PIMA-ODA, L1, or IMB-PIMA-ODA, L2) as a white powder. The reaction conversion yield exceeds 90 %.

**3) Synthesis of AA20%-PIMA-OCA80% and IA20%-PIMA-OCA80%.** In a 100 mL round-bottom flask equipped with stirring bar, PIMA (1g, 6.64 mmol of monomer units) was dissolved in 5 mL of dimethylformamide (DMF). Then, either N, N-dimethylaminopropylamine (163 μL, 1.33 mmol), or 1-(3-Aminopropyl) imidazole (162.5 mg, 1.33 mmol) dissolved in 1 mL of DMF was added dropwise to the PIMA solution. The reaction mixture was stirred for 30 min at 60 °C, followed by the addition of octylamine (OCA, 881 μL, 5.33 mmol) dissolved in 2 mL of DMF, then further left stirring at 60 °C overnight. After that, the solvent was evaporated under vacuum and the residue was dissolved in 5 mL of CHCl<sub>3</sub>. This chloroform solution was added dropwise to a 500 mL flask containing 200 mL of ethyl acetate under vigorous stirring, resulting in precipitation of the product. The turbid solution was passed through a filter paper, collecting the solid white residue. After drying, the above precipitation process using ethyl acetate was repeated one more time, to provide the final product as a white powder; the reaction yield was ~80%.

**4) Synthesis of L2: AMB20%-PIMA-OCA80% and L5: IMB20%-PIMA-OCA80%.** The bromide salt transformation was carried out following the protocol described above for the ODA-containing polymers. Each of the compounds obtained in the last step (step 3) was dissolved in THF (10 mL) in a pressure vessel, then 5 mL of bromoethane was added. The reaction mixture was then stirred overnight at 70 °C. The solvent was evaporated and the residue was dissolved in 4 mL of CHCl<sub>3</sub>, followed by precipitation using excess ethyl acetate under vigorous stirring. The white solid precipitate was collected and dried using a vacuum oven. These steps yielded the products L2 and L5 as white powders, with a reaction yield of ~90% for both compounds.

**5) Synthesis of L3: AMB40%-PIMA-OCA60% and L6: IMB40%PIMA-OCA60%.** The synthetic protocol and purification process were very similar to the synthesis of **L2** and **L5** described above. We first synthesized AM40%-PIMA-OCA60% or IM40%-PIMA-OCA60%, by adjusting the molar ratios of the amine nucleophiles with respect to the PIMA monomer concentration. Following purification, the product was reacted with bromoethane using a pressure vessel as described above. We should note that the polysalt products with 40% of ammonium or imidazolium, namely, AMB40%-PIMA-OCA60%, **L3**, or IMB40%-PIMA-OCA60%, **L6**, were not readily soluble in  $\text{CHCl}_3$  or THF. Instead EtOH was used to dissolve the polymer and implement the purification process.

### **Growth of CsPbBr<sub>3</sub> PQDs**

The CsPbBr<sub>3</sub> PQDs were grown using the hot injection method reported by Kovalenko and coworkers.<sup>3</sup> A few minor modifications have been introduced to allow the implementation of a larger scale reaction. We first prepared a stock solution of Cs-oleate precursor. Briefly, Cs<sub>2</sub>CO<sub>3</sub> (0.814 g, 2.5 mmol), OA (2.5 mL) and octadecene (ODE, 40 mL) were loaded into a 100 mL three-neck round bottom flask equipped with a stir bar. After degassing under vacuum at 120 °C for 1 h, the atmosphere was switched to nitrogen and the content was further heated to 150 °C until the solution becomes clear. The resulting Cs-oleate was stored under nitrogen atmosphere at room temperature. Separately, PbBr<sub>2</sub> (0.2 g, 0.54 mmol) and 15 mL of ODE were mixed in a 50 mL three-neck round bottom flask (equipped with stir bar) and degassed under vacuum for 30 min at 100 °C, while stirring. Aliquots of OLA (1.5 mL) and OA (1.5 mL) were added to the PbBr<sub>2</sub> and ODE mixture through a syringe under N<sub>2</sub> atmosphere. Once PbBr<sub>2</sub> was completely dissolved, the mixture was heated to 170 °C under nitrogen atmosphere followed by rapid injection of 1.2 mL of Cs-oleate stock solution (preheated to 100 °C). After letting the reaction proceed for 5 s, the flask was immersed in an ice bath to arrest the growth. The solution color turned from light yellow to light green over a period of few minutes. We should note that pre-heating the precursor solution to 100 °C prior to injection is critical, because Cs-oleate tends to precipitate out of ODE at room temperature. The growth solution of CsPbBr<sub>3</sub> QDs was first centrifuged at 3600 RPM for 5 min, yielding a precipitate which was retrieved and dispersed in 5 mL of hexane with sonication. One additional round of centrifugation at 3600 RPM for 5 min was applied to remove aggregates. The clear supernatant containing the PQDs was collected. A UV-Vis absorption spectrum was acquired and used to determine the PQDs concentration, using the molar absorption coefficient data reported by Hens and co-workers.<sup>4</sup> Typically, the PQD concentration of the stock dispersion was adjusted to ~5 μM and stored until further use.

### **Ligand Exchange Using L1(AMB20%-PIMA-80%ODA) or L4(IMB20%-PIMA-80%ODA)**

First, ~15 mg of **L1** or **L4** were dissolved in 400 μL of toluene using 1.7 mL of Eppendorf tube. The resulting solution was mixed with an aliquot of CsPbBr<sub>3</sub> PQD stock dispersion (~5 μM, 200 μL) in a 10 mL scintillation vial. After sonication for ~3–5 min, ~2 mL of ethanol (i.e., in excess)

was used to precipitate the newly coated PQDs. The turbid dispersion was subjected to one round of centrifugation (3500 RPM, 5 min), and then the PQD pellet was retrieved and dispersed in toluene (400  $\mu$ L). The resulting dispersions of L1- and L4-stabilized PQDs were used to carry out additional characterization experiments.

### **Ligand Exchange Using L2 and L3 (AMB-PIMA-OCA) or L5 and L6 (IMB-PIMA-OCA)**

Two configurations were used to carry out the ligand exchange: one and two-phase reactions.

**One-phase ligand exchange reaction:** First, AMB-PIMA-OCA (10 mg) or IMB-PIMA-OCA (10 mg) were dissolved in THF (600  $\mu$ L). The ligand solution was mixed with 200  $\mu$ L of CsPbBr<sub>3</sub> stock dispersion in hexane (5  $\mu$ M), to yield a bright green clear solution. After sonication for 2 min, excess hexane (~3 mL) was added to precipitate the newly-capped PQDs. One round of centrifugation (3500 RPM, 5 min) was applied yielding a pellet of the PQDs and a clear supernatant. The green pellet was dispersed in 400  $\mu$ L of THF, resulting in a clear bright dispersion. The PQD dispersion was subjected to one additional round of precipitation using ethyl acetate (2 mL). The collected PQD pellet was then dispersed in the desired polar solvent (e.g., ethanol or methanol) and used for further characterization. The polymer ligands presenting 40% AMB or IMB and 60% OCA are not completely soluble in THF. Thus, a mixture of THF and ethanol (400  $\mu$ L: 200  $\mu$ L) was used to solubilize the ligand instead. Mixing the ligand with the PQD stock dispersion yielded a turbid mixture. Adding 1 mL of hexane promoted precipitation of the PQDs, which were centrifuged (3500 RPM, 5 min) into a pellet. The pellet was retrieved and transferred to a THF and ethanol mixture (200  $\mu$ L: 100  $\mu$ L), then 1 mL of ethyl acetate was added to wash the dispersion. These steps yielded PQDs that were readily dispersible in ethanol or methanol. We note that trace amount of aggregates produced during the ligand exchange using L3 and L6 can be removed by applying another round of centrifugation (3500 RPM, 5 min).

**Two-phase ligand exchange reaction:** 200  $\mu$ L aliquot of CsPbBr<sub>3</sub> stock dispersion (5  $\mu$ M) was diluted with hexane to a final volume of 500  $\mu$ L and loaded in a 10 mL scintillation vial containing AMB-PIMA-OCA (10 mg) or IMB-PIMA-OCA (10 mg) dissolved in 500  $\mu$ L of MeOH. A clear two-phase solution was formed, with the green top layer containing the PQDs and the bottom layer containing the ligands. Stirring and sonication for ~ 3–5 min yielded a turbid two-phase mixture. 500  $\mu$ L of hexane was added and one round of centrifugation (3600 RPM, 5 min) was applied to promote the phase separation. The top hexane layer was removed using a pipette and the methanol dispersion of the PQDs was retrieved and preserved until further use.

### **NMR Sample Preparation**

To prepare a sample of the native OLA/OA–PQDs, 500  $\mu$ L aliquot of a stock dispersion was precipitated using ethyl acetate (2 mL) followed by one round of centrifugation (3500 RPM, 5 min). After drying the retrieved pellet under vacuum, the nanocrystals were dispersed in 500  $\mu$ L

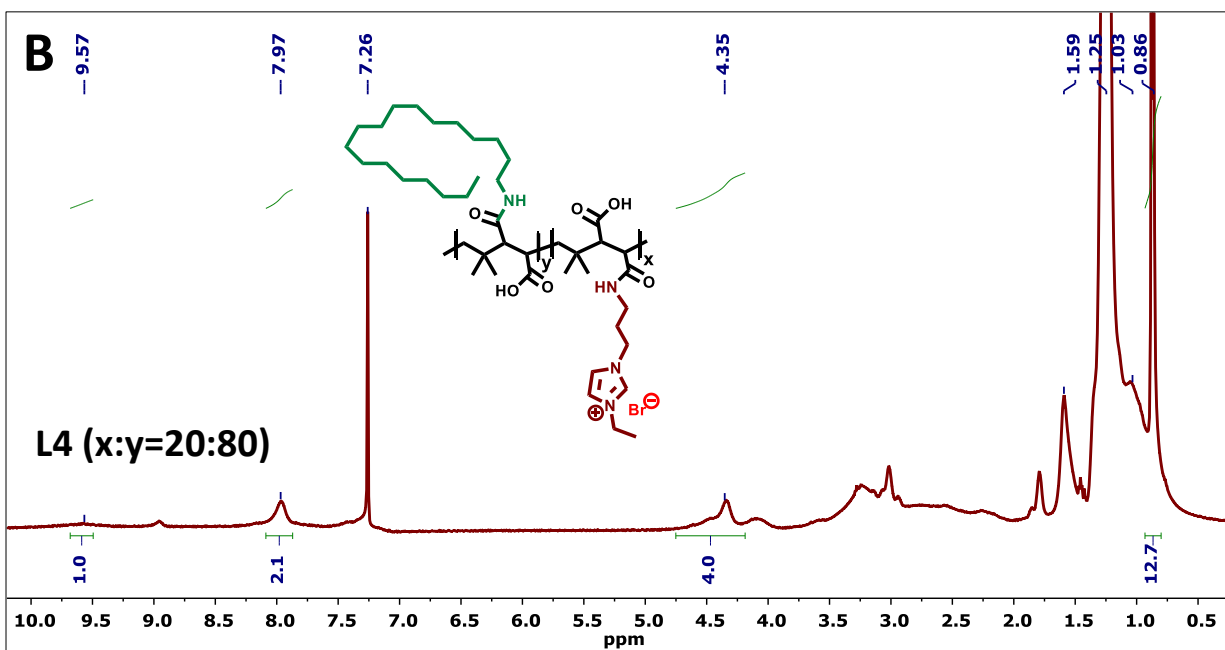
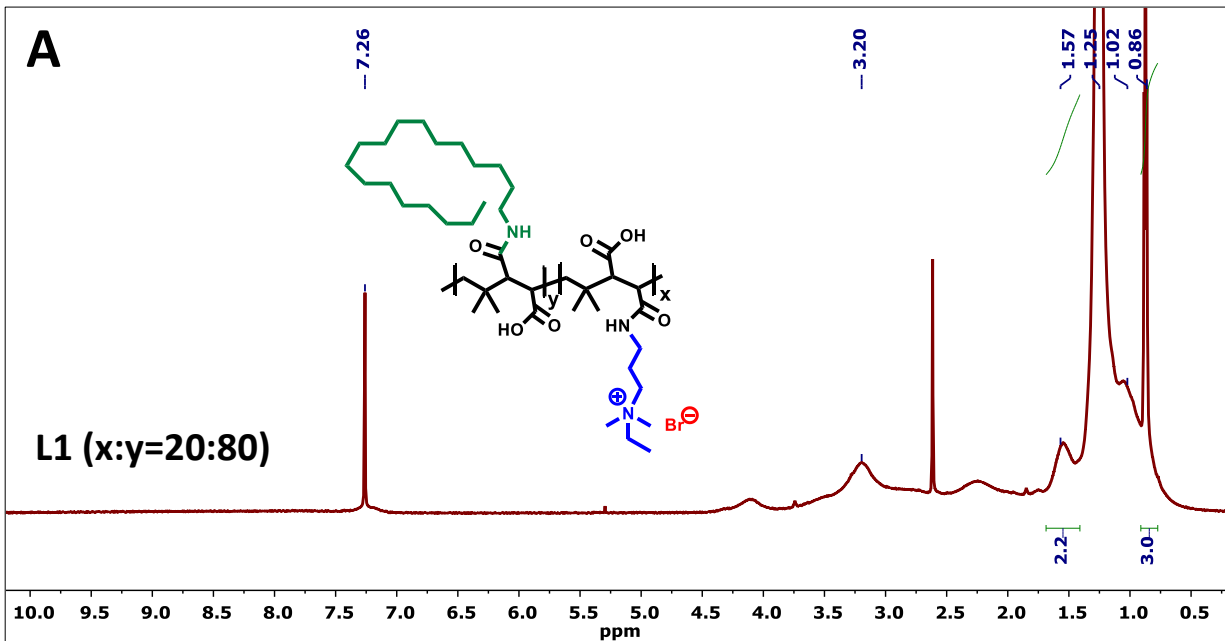
of  $\text{CDCl}_3$  and then loaded into an NMR tube for spectral collection. The NMR samples of PQDs ligand substituted with **L4** or **L5** were prepared in a 500- $\mu\text{L}$  volume following the same ligand exchange protocols described above, except that  $\text{CHCl}_3$  was substituted with  $\text{CDCl}_3$  when preparing the final dispersions of the nanocrystals. In particular, PQDs stabilized with **L4** were subjected to two rounds of washing with ethanol, while PQDs capped with **L5** were precipitated twice with ethyl acetate, before drying the pellet and re-dispersion in  $\text{CDCl}_3$ . NMR spectra were collected using 512 scans on average.

## Stability Tests

**1) EtOH wash test applied to L1- and L4-stabilized PQDs.** Toluene Dispersions of PQDs (400  $\mu\text{L}$ , 2.5  $\mu\text{M}$ ) ligated with **L1** (AMB20%-PIMA-ODA) or **L4** (IMB20%-PIMA-ODA) were transferred to 7-mL vials. Then, 2 mL of EtOH were added to the two vials to precipitate the nanocrystals, followed by centrifugation (3500 RPM, 5 min). The obtained precipitates were re-dispersed in toluene and then used to collect white light and fluorescence images, along with the absorption and PL spectra. The procedure was repeated four more times but at higher RPM (12000 RPM, 5 min) was applied each time to precipitate the PQDs, and images and spectra were collected after each round. The data from these dispersions were compared to those measured from a dispersion of OLA/OA-PQDs, which was subjected to only one round of precipitation using ethanol.

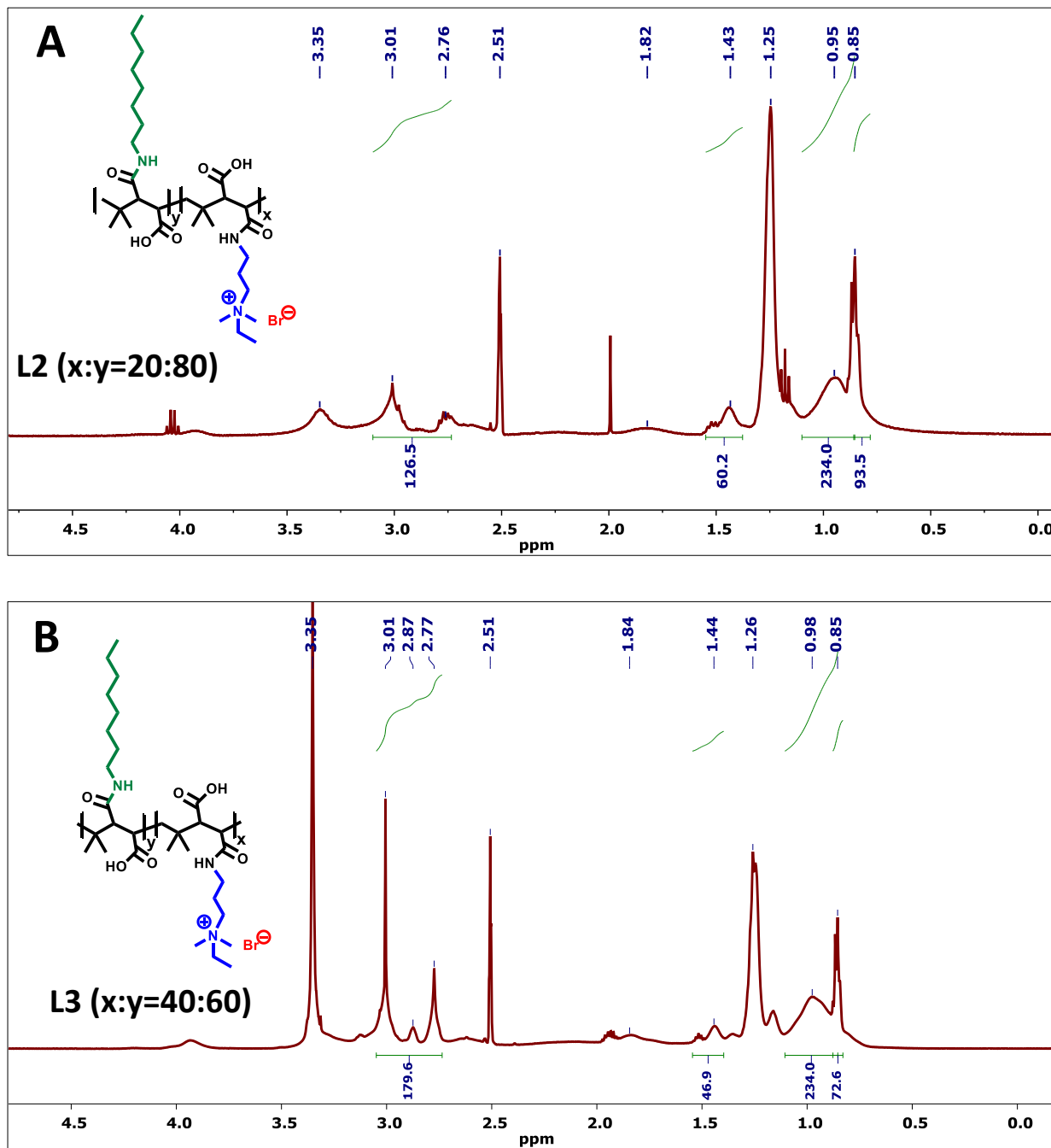
**2) Water-resistance test applied to L1- and L4-stabilized PQDs.** 200  $\mu\text{L}$  of OLA/OA-PQDs stock dispersion in hexane was dried under  $\text{N}_2$  flow, and then dispersed in 2 mL of toluene. Similarly, L1-PQDs and L4-PQDs (400  $\mu\text{L}$ ) were diluted in 2-mL of toluene. Those PQD dispersions were slowly added to 7-mL vials containing 2 mL of DI water. The vials containing the mixtures were kept static at room temperature and white light exposure. Images of those dispersions were taken every 24 h under both white light exposure and UV light excitation (fluorescence mode).

**3) Long term colloidal stability test applied to L2-, L3-, L5-, and L6-stabilized PQDs.** All the samples used for long-term colloidal stability tests were prepared using one-phase ligand exchange reaction. Following ligand substitution, 200  $\mu\text{L}$  of AMB-PIMA-OCA (**L2** or **L3**)– and IMB-PIMA-OCA (**L5** and **L6**)–PQDs dispersed in ethanol or methanol were loaded onto 2 mL glass vials. Then 300  $\mu\text{L}$  of EtOH or MeOH were added to bring the total volume to  $\sim 500$   $\mu\text{L}$ . The vials were sealed and stored under room light and temperature conditions. Fluorescence images of the dispersions under UV illumination (using a hand-held UV lamp, excitation at 365 nm) were taken every month.

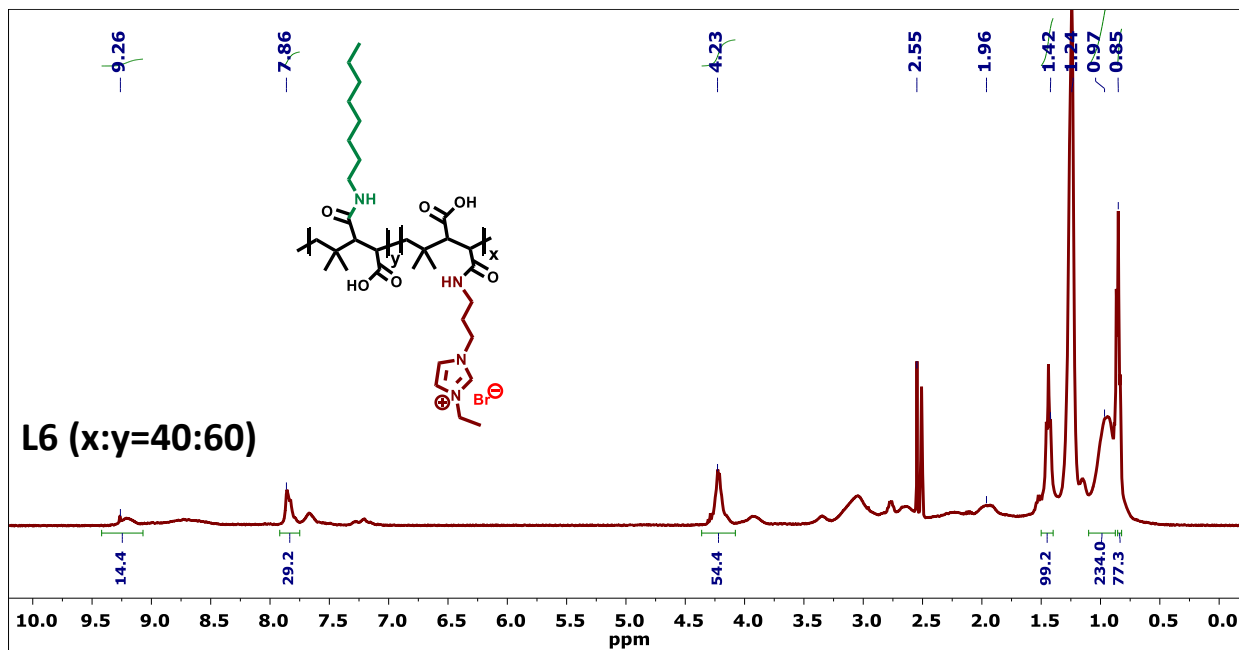


**Figure S1:** (A,B)  $^1\text{H}$  NMR spectra collected from solutions of **L1** and **L4**, respectively.  $\text{CDCl}_3$  was used as solvent.

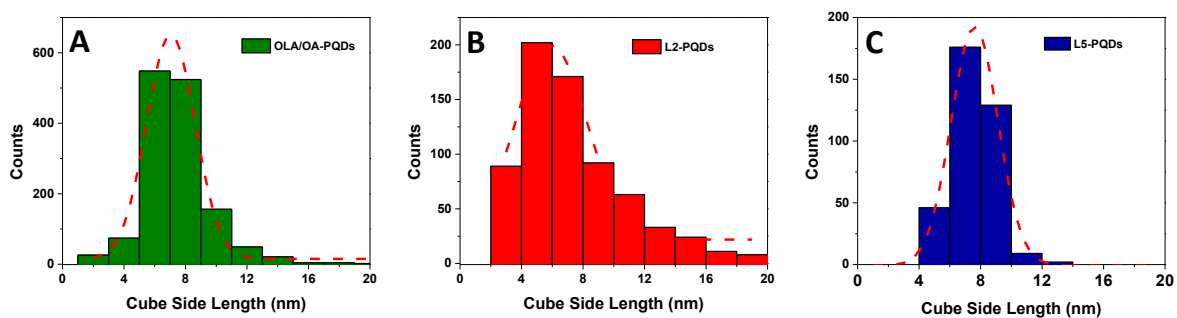




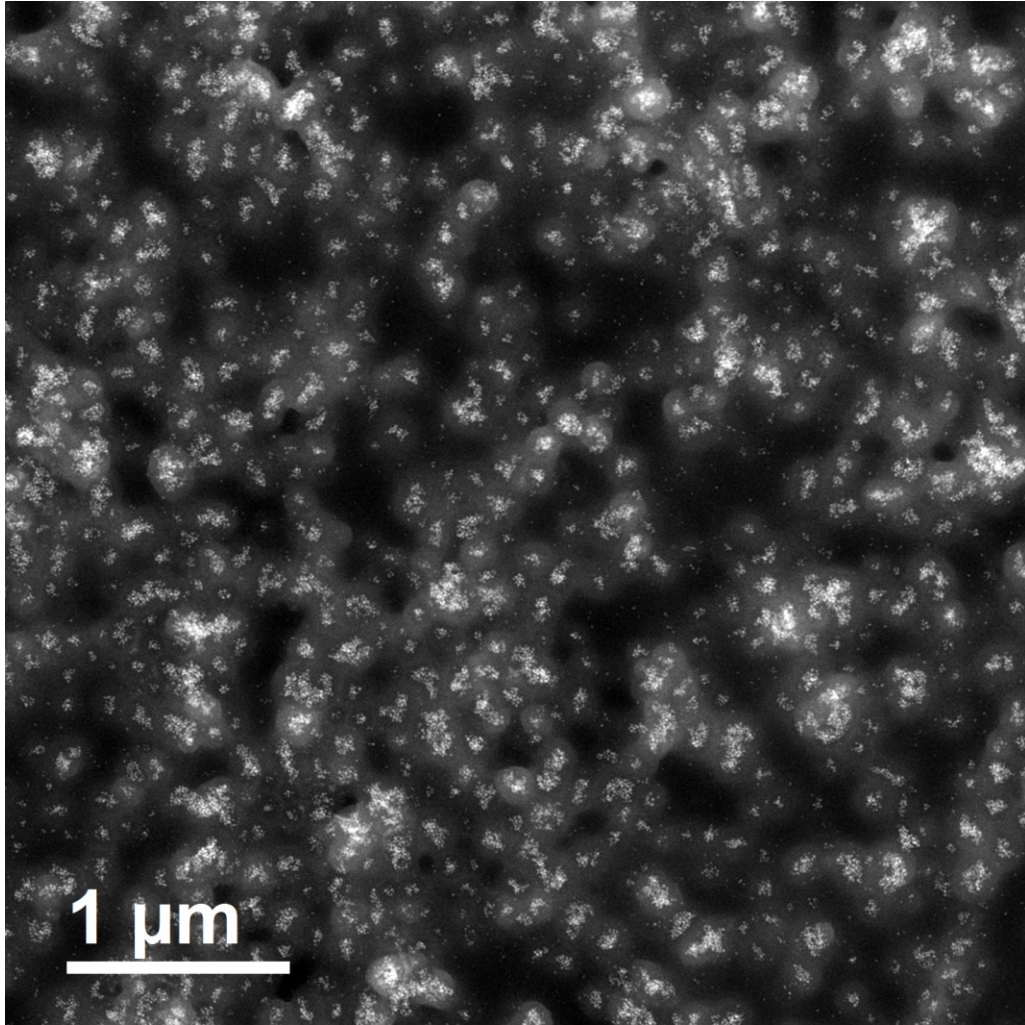
**Figure S2:** (A)  $^1\text{H}$  NMR spectrum collected from L2. (B)  $^1\text{H}$  NMR spectrum of L3. The polymers were dissolved in DMSO- $d_6$ .



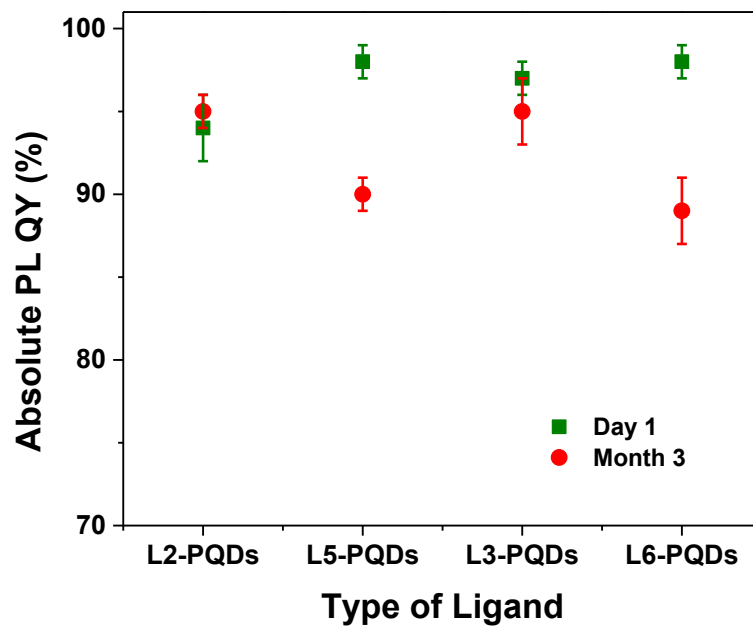
**Figure S3:** <sup>1</sup>H NMR spectrum collected from L6. The polymer was dissolved in DMSO-d<sub>6</sub>.



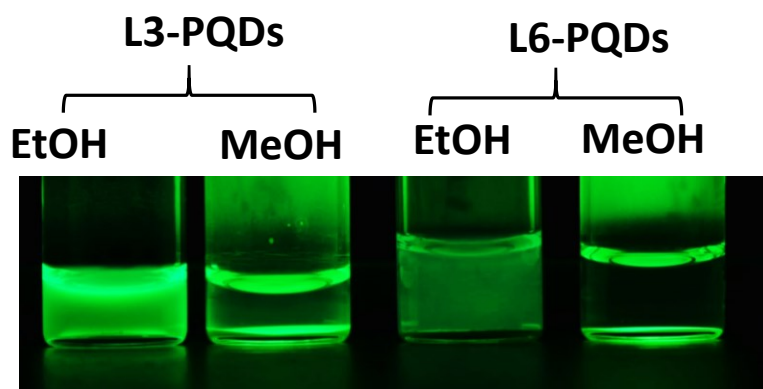
**Figure S4:** Histograms of the cube edge size distribution extracted from TEM data for OLA/OA-PQDs (A), L2-PQDs (B) and L5-PQDs (C). The TEM images are shown in the main text Figure 3.



**Figure S5:** A larger view STEM image acquired from L5-PQDs showing patterns of small nanocrystal clustering.



**Figure S6:** Absolute PLQY values measured for different dispersions of PQDs, surface stabilized with OCA-modified polymers presenting different fractions of AMB and IMB salt groups. Values are reported for freshly prepared samples and after 3 months of storage.



**Figure S7:** Fluorescence image of L3- and L6-capped PQD dispersions in ethanol and methanol, after 12 months of storage.

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