Supporting Information

**Designed Long-Lived Emission from CdSe Quantum Dots through Reversible Electronic Energy Transfer with a Surface-Bound Chromophore**

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1. Materials and methods

1a. Chemicals and solvents
Cadmium nitrate tetrahydrate (Cd(NO\textsubscript{3})\textsubscript{2}·4H\textsubscript{2}O, 98%), selenium powder (Se, 99.5%, 100 mesh), sulfur (elemental S, 99.98%, powder), \textit{n}-trioctylphosphine oxide (TOPO, 90%), \textit{n}-trioctylphosphine (TOP, 97%), \textit{n}-hexadecylamine (HDA, 98%), diethylzinc solution (1.0 M in hexane), hexamethyldisilathiane (synthesis grade), deoxycholic acid (≥99%, HPLC), sodium deoxycholate (≥97%), were purchased from Sigma Aldrich and used without any further purification. 1-Pyrenecarboxylic acid (1-PCA, >97.0%, GC) was purchased from TCI America and recrystallized from ethanol. Synthetic grade heptane, acetone, ethanol and methanol were purchased from Alfa Aesar. Spectrophotometric grade chloroform and heptane were purchased from Alfa Aesar and used without any further purification.

1b. Synthesis and characterization of CdSe Quantum Dots (QDs)
Four different batches of CdSe Core QDs were synthesized according to the procedure reported by Maitra and coworkers.\textsuperscript{[S1]} Briefly, a solution of Cd(NO\textsubscript{3})\textsubscript{2} in MeOH (30 mL, 50 mM) was added to a methanolic solution of sodium deoxycholate (180 mL, 25 mM) and stirred. A fine precipitate of cadmium deoxycholate started appearing within a minute and the solution became viscous. After 15 minutes the formation of a gel had been observed and the mixture was stirred for another 45 minutes. The white precipitate was filtered off, washed with cold MeOH (2 × 5 mL) and dried overnight under vacuum. Successively, cadmium deoxycholate (0.049 g, 0.05 mmol), deoxycholic acid (0.128 g, 0.32 mmol), HDA (1 g, 4.14 mmol) and TOPO (1 g, 2.6 mmol) were placed in a three-neck round bottom flask, degassed under vacuum and heated up to 300°C under argon flow. TOP-Se solution (0.5 mL), prepared in a dry box by dissolving selenium powder (0.039 g, 0.5 mmol) in TOP (0.5 mL, 1.1 mmol), were quickly injected to the cadmium precursor solution. After the injection, the growth temperature was kept at 290°C. Aliquots were taken during the synthesis to check the size of growing nanocrystals. When the desired size had been reached, the reaction was stopped by cooling the mixture to room temperature. Finally, QDs were purified by precipitation in a mixture of acetone and MeOH (5:1, v/v). The precipitate was isolated by decantation and centrifugation at least three times and, ultimately, stored in heptane. The diameter and the molar absorption coefficient ($\varepsilon$) of each QD sample were estimated from the lowest exciton absorption peak (Fig. S1) according to a previously reported method.\textsuperscript{[S2]} The concentration of the nanocrystals in solution was calculated with Beer’s law from the absorbance and $\varepsilon$ values. The size of the QDs was confirmed by TEM measurements (Fig. S2): a drop of QD
solution was deposited on a Formvar resin film supported on conventional copper microgrids, the sample was dried under vacuum, and was imaged with a Philips CM 100 transmission electron microscope operating at 80 kV.

Core-shell CdSe-ZnS QDs were prepared by coating a CdSe Core sample with a ZnS shell using the one-time-precursor-injection approach. The resulting nanocrystals were covered with TOPO, TOP and HDA as surfactants to prevent their aggregation.

1c. Surface functionalization with 1-pyrenecarboxylic acid (1-PCA)
QDs and 1-PCA (1:3000, QD:1-PCA equivalent ratio) were mixed in heptane; then the resulting mixture was stirred for 1 hour at rt. Within this time a white precipitate appeared, consisting of the displaced surfactants that are not soluble in heptane (i.e., deoxycholic acid and HDA), thus indicating that surface modification has occurred. The mixture was then filtered to remove both precipitated ligands and undissolved 1-PCA. As 1-PCA is nearly insoluble in heptane, the pyrenyl species that become dissolved in this solvent in the presence of the QDs are necessarily associated with the nanocrystals.

The average number of 1-PCA ligands per nanoparticle was estimated from absorption spectra, assuming that the molar absorption coefficients of 1-PCA and the QD are additive in the hybrids. The upper limit of pyrenyl ligands not bound to the QDs corresponds to the solubility of 1-PCA in heptane, which was estimated to be 1.5×10^{-6} M by absorption spectroscopy at rt. This number was utilized to assess the experimental error on the average number of 1-PCA ligands per QD. Under the experimental conditions employed, unbound 1-PCA species are less than 8% of the total number of pyrenyl ligands in the worst case.

It can be noticed (Tables S2-S3) that CdSe-1 and CdSe-2 QDs exhibit slightly decreased luminescence lifetimes in the 100-ns range upon functionalization with 1-PCA. It is commonly accepted[55,56] that the replacement of the native caps with new ligands in core-only QDs can lead to an incomplete passivation of the surface, thus causing an increased contribution of trap states and a consequent decrease of luminescence quantum yield and lifetime. Such an interpretation is confirmed by the fact that the exchange with 1-PCA does not affect the lifetime of core-shell CdSe-ZnS QDs (Table S6). Our results (Tables S2-S5) show that the lifetime decrease is inversely proportional to the QD size, in keeping with the fact that surfaces with higher curvature are expected to be more defective and reactive.[57] A detailed analysis of these effects is beyond the scope of the present work. The study of ligand exchange reactions and consequent purification procedures, and their effect on the photophysical properties of the nanocrystals, is indeed a primary topic of current research.[58]

1d. Sample preparation
Samples were prepared by diluting a stock solution of both 1-PCA decorated CdSe QDs and pyrene-free CdSe QDs with spectrophotometric grade heptane to reach the desired
absorbance/concentration value. For the luminescence experiments, the concentration of the samples in solution was comprised between $1 \times 10^{-7}$ and $1 \times 10^{-6}$ M, and the absorbance in the first exciton peak was below 0.1 to avoid inner filtering effects. For direct comparison of samples, each solution of 1-PCA decorated QDs was prepared with the same absorption value at the first excitonic peak with respect to the corresponding pyrene-free variant. Samples for transient absorption experiments were prepared in 1 cm quartz cuvettes, with a minimum optical density of 0.4 at the excitation wavelength ($\lambda = 465$ nm). All the samples were rigorously deaerated at least three times through freeze-pump-thaw cycles and the cells were blowtorch sealed before experiments.

1e. Absorption and luminescence measurements

Electronic absorption spectra in the 210-800 nm range were recorded on a Varian Cary 300 spectrophotometer. The precision on the wavelength values was ± 1 nm. Luminescence emission, both in fluorescence and phosphorescence mode, in the 250-800 nm range was recorded with a Perkin Elmer LS-55 spectrofluorimeter. The precision on the wavelength values was ± 1 nm. Luminescence quantum yields were determined with the optically dilute method using fluorescein ($\Phi = 0.97$ in 0.1 M NaOH) or [Ru(bpy)$_3$]Cl$_2$ (bpy = 2,2'-bipyridine, $\Phi = 0.027$ in H$_2$O) as standards.$^{[59]}$ Lifetime measurements in the 500 ns time range were carried out at room temperature with an Edinburgh Instruments FLS-920 time-correlated single photon counting (TCSPC) spectrometer. A 405 nm pulsed diode laser (Picoquant) was used as the light source, and the detector was a Hamamatsu R928P photomultiplier cooled to −20°C and suitably amplified. All the experiments were carried out at room temperature in 1 cm quartz cuvettes. Deoxygenation was performed by at least four freeze-pump-thaw cycles.

1f. Streak Camera Experiments

Two different streak camera set-ups were utilized to allow recording across the necessary wide time range.

**Picosecond set-up:** A Ti:sapphire laser system emitting pulses of 0.6 mJ and 30 fs at 800 nm and 1 kHz pulse repetition rate (Femtopower Compact Pro) with Light Conversion Topas-C optical parametric generator and frequency mixers was used to excite the samples at chosen wavelength. All excited-state lifetimes were obtained by using depolarized excitation light. The highest pulse energies used to excite fluorescence did not exceed 100 nJ and the average power of excitation beam was 0.1 mW at a pulse repetition rate of 1 kHz focused into a spot with a diameter of 0.1 mm in the 10 mm-long fused-silica cell. The fluorescence emitted in the forward direction was collected by reflective optics and focused with a spherical mirror onto the input slit of a spectrograph (Chromex 250) coupled to a streak camera (Hamamatsu 5680 equipped with a fast single sweep unit M5676, temporal resolution 2 ps). Convolution of a rectangular streak camera slit in the sweep range of 250 ps with electronic jitter of the streak
camera trigger pulse provided a Gaussian (over four decades) temporal apparatus function with 
a full width at half-maximum of 20 ps.

**Nanosecond set-up:** A tunable nanosecond laser (5 ns, 135 mJ @355 nm from Nd:YAG 
amplified laser pumping OPO, 10 Hz, Ekspla model NT342B-10-WW); produced tunable 
excitation pulses in the range 410-2300 nm. Light signals were analyzed by a spectrograph 
(Princeton Instruments Acton model SP2300) coupled with a high dynamic range streak camera 
(Hamamatsu C7700).
2. Characterization of the QD and 1-PCA components

**Figure S1.** Normalized electronic absorption (left) and emission (right, $\lambda_{\text{exc}} = 450$ nm) spectra of CdSe-1 (green lines), CdSe-2 (yellow lines), CdSe-3 (orange lines) and CdSe-4 (red lines) in air-equilibrated heptane at rt.

**Table S1.** Spectroscopic and morphological properties of CdSe-1, CdSe-2, CdSe-3 and CdSe-4 QDs.

| QD Sample | Diameter (nm)$^{[a]}$ | Absorption$^{[b]}$ | Emission |
|-----------|------------------------|---------------------|-----------|
|           |                        | $\lambda_{\text{max}}$ (nm) | $\varepsilon$ (L mol$^{-1}$cm$^{-1}$)$^{[a]}$ | $\lambda_{\text{max}}$ (nm) |
| CdSe-1    | 2.61                   | 524                 | 75000     | 535       |
| CdSe-2    | 3.26                   | 560                 | 134000    | 573       |
| CdSe-3    | 4.02                   | 586                 | 234500    | 596       |
| CdSe-4    | 5.72                   | 622                 | 597000    | 636       |

$^{[a]}$ Determined from the position of the lowest energy absorption peak according to ref. [S2]. $^{[b]}$ Lowest energy exciton peak.
Figure S2. TEM micrographs of CdSe-1 (a), CdSe-2 (b), CdSe-3 (c) and CdSe-4 (d) QDs.
**Figure S3.** Absorption (solid line, left scale) and fluorescence (\(\lambda_{\text{exc}} = 340\) nm, dashed line) spectra of 1-PCA in toluene at rt. The dotted line is the phosphorescence spectrum of 1-PCA in THF rigid matrix at 77 K (\(\lambda_{\text{exc}} = 340\) nm, delay 0.05 ms, gate 9.95 ms).

**Figure S4.** Phosphorescence decay of 1-PCA in THF rigid matrix at 77 K, monitored at 600 nm upon excitation at 350 nm. The solid line is the fitting to a single exponential function with \(\tau = 274\) ms (\(\chi^2 = 1.2\)).
3. Steady state and time resolved absorption and luminescence data

3a. CdSe-1 samples

Figure S5. Normalized absorption (solid lines) and emission spectra (dashed lines) of CdSe-1 QDs (green) and free 1-PCA (blue) recorded, respectively, in heptane ($\lambda_{\text{exc}} = 470$ nm) and in THF ($\lambda_{\text{exc}} = 340$ nm) at rt.

Figure S6. Absorption (full lines, left scale) and luminescence (dashed lines, right scale; $\lambda_{\text{exc}} = 470$ nm) spectra of CdSe-1 (grey traces) and CdSe-1@1-PCA (purple traces) QDs in air equilibrated heptane at rt. The concentration of the two samples is $1.1 \times 10^{-6}$ M and the average number of 1-PCA units per QD is 68±2.
Figure S7. Luminescence decay of CdSe-1 (grey) and CdSe1@1-PCA (purple) QDs monitored at 540 nm in deoxygenated heptane at rt, measured by time-correlated single-photon counting (log plot, $\lambda_{\text{exc}} = 405$ nm).

Figure S8. Luminescence decay of CdSe-1 (grey) and CdSe1@1-PCA (purple) QDs monitored at 540 nm in air equilibrated heptane at rt, measured by time-correlated single-photon counting (log plot, $\lambda_{\text{exc}} = 405$ nm).
### Table S2. Photophysical properties of CdSe-1 QDs with and without 1-PCA.

| Sample                | Air-equilibrated heptane | Deaerated heptane |
|-----------------------|--------------------------|-------------------|
|                       | $\Phi$ | $\tau (F)^{[a]}$ | $\chi^2$ | $\Phi$ | $\tau (F)^{[a]}$ | $\chi^2$ |
| CdSe-1                | 0.18  | 8 ns (13%)        | 1.15    | 0.18  | 6 ns (12 %)      | 1.15    |
|                       |       | 38 ns (55%)       |         |       | 37 ns (53 %)     |         |
|                       |       | 133 ns (32%)      |         |       | 133 ns (35 %)    |         |
| CdSe-1 @1-PCA         | 0.04  | 4 ns (14%)        | 1.1     | 0.04  | 5 ns (13 %)      | 1.1     |
|                       |       | 22 ns (60%)       |         |       | 23 ns (53 %)     |         |
|                       |       | 70 ns (26%)       |         |       | 73 ns (34 %)     |         |

[a] Fractional contribution to the overall decay.

#### 3b. CdSe-2 samples

![Normalized absorption (solid lines) and emission spectra (dashed lines) of CdSe-2 QDs (yellow) and free 1-PCA (blue) recorded, respectively, in heptane ($\lambda_{exc} = 500$ nm) and in THF ($\lambda_{exc} = 340$ nm) at rt.](#)

**Figure S9.** Normalized absorption (solid lines) and emission spectra (dashed lines) of CdSe-2 QDs (yellow) and free 1-PCA (blue) recorded, respectively, in heptane ($\lambda_{exc} = 500$ nm) and in THF ($\lambda_{exc} = 340$ nm) at rt.
**Figure S10.** Absorption (full lines, left scale) and luminescence (dashed lines, right scale; $\lambda_{\text{exc}} = 500$ nm) spectra of CdSe-2 (grey) and CdSe-2@1-PCA (blue) QDs in air equilibrated heptane at rt. The concentration of the two samples is $3.7 \times 10^{-7}$ M and the average number of 1-PCA units per QD is $80 \pm 4$.

**Figure S11.** Luminescence decay of CdSe-2 (grey) and CdSe2@1-PCA (blue) QDs monitored at 580 nm in deoxygenated heptane at rt, measured by time-correlated single-photon counting (log plot, $\lambda_{\text{exc}} = 405$ nm).
**Figure S12.** Luminescence spectra of optically matched solutions of CdSe-2@1-PCA (blue) and CdSe-2 (grey) recorded in deoxygenated heptane at different delay times (full lines, 40 µs; dashed lines, 80 µs; dotted lines, 100 µs) upon pulsed excitation at 500 nm.

**Figure S13.** Luminescence decay of CdSe-2 (grey) and 1-PCA decorated CdSe-2 (blue) QDs in heptane ($\lambda_{\text{exc}} = 465$ nm) recorded by streak camera.
Figure S14. Luminescence decay of CdSe-2 (grey) and CdSe2@1-PCA (blue) QDs monitored at 580 nm in air equilibrated heptane at rt, measured by time-correlated single-photon counting (log plot, λ_{exc} = 405 nm).

Figure S15. Luminescence spectra of optically matched solutions of CdSe-2@1-PCA (blue) and CdSe-2 (grey) in air equilibrated heptane, recorded 40 µs after the pulsed excitation at 500 nm.
Table S3. Photophysical properties of CdSe-2 QDs with and without 1-PCA.

| Sample          | Air-equilibrated heptane | Deaerated heptane |
|-----------------|--------------------------|-------------------|
|                 | $\Phi$ | $\tau (F)^{[a]}$ | $\chi^2$ | $\Phi$ | $\tau (F)^{[a]}$ | $\chi^2$ |
| CdSe-2          | 0.16  | 9 ns (12%)       | 1.08     | 0.16  | 9 ns (12%)       | 1.07     |
|                 |       | 30 ns (64%)      |          |       | 30 ns (66%)      |          |
|                 |       | 76 ns (24%)      |          |       | 79 ns (21%)      |          |
| CdSe-2 @1-PCA   | 0.06  | 6 ns (16%)       | 1.2      | 0.06  | 0.038; 14; 36; 130 ns (98.4%) |          |
|                 |       | 22 ns (64%)      |          |       | 1.1; 18 $\mu$s (0.1%) |          |
|                 |       | 66 ns (20%)      |          |       | 330 $\mu$s (1.5%)$^{[b]}$ |          |

[a] Fractional contribution to the overall decay. [b] From fitting streak camera data (multiple time windows).

3c. CdSe-3 samples

Figure S16. Normalized absorption (solid lines) and emission spectra (dashed lines) of CdSe-3 QDs (orange) and free 1-PCA (blue) recorded, respectively, in heptane ($\lambda_{exc} = 500$ nm) and in THF ($\lambda_{exc} = 340$ nm) at rt.
**Table S4.** Photophysical properties of CdSe-3 QDs with and without 1-PCA.

| Sample               | Air-equilibrated heptane | Deaerated heptane             |
|----------------------|--------------------------|--------------------------------|
|                      | \( \Phi \) | \( \tau (F)^{[a]} \) | \( \chi^2 \) | \( \Phi \) | \( \tau (F)^{[a]} \) | \( \chi^2 \) |
| CdSe-3               | 0.57       | 16 ns (18%)               | 1.15 | 0.57       | 17 ns (20 %)               | 1.07 |
|                      |            | 32 ns (72%)               |       |            | 33 ns (78 %)               |     |
|                      |            | 84 ns (10%)               |       |            | 89 ns (10 %)               |     |
| CdSe-3 @1-PCA        | 0.44       | 16 ns (25%)               | 1.17 | 0.46       | 0.080; 19; 64; 260 ns (98%) | – |
|                      |            | 34 ns (64%)               |       |            | 4.1; 22 µs (0.2%)          |     |
|                      |            | 104 ns (11%)              |       |            | 210 µs (1.8%)^{[b]}        |     |

[a] Fractional contribution to the overall decay. [b] From fitting streak camera data (multiple time windows).

**3d. CdSe-4 samples**

![Normalized absorption and emission spectra of CdSe-4 QDs and free 1-PCA](image)

**Figure S17.** Normalized absorption (solid lines) and emission spectra (dashed lines) of CdSe-4 QDs (red) and free 1-PCA (blue) recorded, respectively, in heptane (\( \lambda_{exc} = 500 \text{ nm} \)) and in THF (\( \lambda_{exc} = 340 \text{ nm} \)) at rt.
**Figure S18.** Absorption (full lines, left scale) and luminescence (dashed lines, right scale; \( \lambda_{\text{exc}} = 500 \) nm) spectra of CdSe-4 (grey) and CdSe-4@1-PCA (red) QDs in air equilibrated heptane at rt. The concentration of the two samples is \( 1.0 \times 10^{-7} \) M and the average number of 1-PCA units per QD is 300±15.

**Figure S19.** Luminescence decay of CdSe-4 (grey) and CdSe4@1-PCA (red) QDs monitored at 636 nm in deoxygenated heptane at rt, measured by time-correlated single-photon counting (log plot, \( \lambda_{\text{exc}} = 405 \) nm).
Figure S20. Luminescence decay of CdSe-4 (grey) and CdSe4@1-PCA (red) QDs monitored at 636 nm in air equilibrated heptane at rt, measured by time-correlated single-photon counting (log plot, $\lambda_{\text{exc}} = 405$ nm).

Table S5. Photophysical properties of CdSe-4 QDs with and without 1-PCA.

| Sample            | Air-equilibrated heptane | Deaerated heptane |
|-------------------|--------------------------|-------------------|
|                   | $\Phi$                   | $\tau (F)^{[a]}$ | $\chi^2$ | $\Phi$ | $\tau (F)^{[a]}$ | $\chi^2$ |
| CdSe-4            | 0.03                     | 5 ns (17%)        | 1.06     | 0.03   | 5 ns (18%)        | 1.07     |
|                   |                          | 15 ns (57%)       |          |        | 18 ns (57%)       |          |
|                   |                          | 37 ns (26%)       |          |        | 37 ns (25%)       |          |
| CdSe-4 @1-PCA     | 0.01                     | 5 ns (17%)        | 1.1      | 0.01   | 3 ns (14%)        | 1.1      |
|                   |                          | 15 ns (56%)       |          |        | 14 ns (55%)       |          |
|                   |                          | 41 ns (27%)       |          |        | 40 ns (31%)       |          |

[a] Fractional contribution to the overall decay.
3e. CdSe-ZnS core-shell samples

**Figure S21.** Absorption (full lines, left scale) and luminescence (dashed lines, right scale; $\lambda_{\text{exc}} = 500$ nm) spectra of core-shell QDs composed of a CdSe-3 core with a shell of 2 monolayers of ZnS, with (cyan) and without (grey) 1-PCA in air equilibrated heptane at rt. The concentration of the two samples is $4.8 \times 10^{-7}$ M and the average number of 1-PCA units per QD is 250\(\pm\)4.

**Figure S22.** Luminescence decay of CdSe-ZnS QDs (CdSe-3 core coated with 2 ZnS layers) with (cyan) and without (grey) 1-PCA monitored at 610 nm in air equilibrated heptane at rt, measured by time-correlated single-photon counting (log plot, $\lambda_{\text{exc}} = 405$ nm).
Table S6. Photophysical properties of CdSe-ZnS QDs (CdSe-3 core coated with 2 ZnS layers) with and without 1-PCA.

| Sample                  | Deaerated heptane                              | $\tau$ (F)$^a$ | $\chi^2$ |
|-------------------------|-----------------------------------------------|----------------|----------|
| CdSe-ZnS                |                                               | 6 ns (12%)      | 1.09     |
|                         |                                               | 22 ns (76%)     |          |
|                         |                                               | 62 ns (12%)     |          |
| CdSe-ZnS @1-PCA         |                                               | 5 ns (10%)      | 1.2      |
|                         |                                               | 22 ns (78%)     |          |
|                         |                                               | 62 ns (12%)     |          |

[a] Fractional contribution to the overall decay.

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