Supporting information

Low-Temperature Approach to High-Yield and Reproducible Syntheses of High-Quality Small-Sized PbSe Colloidal Nanocrystals for Photovoltaic Applications

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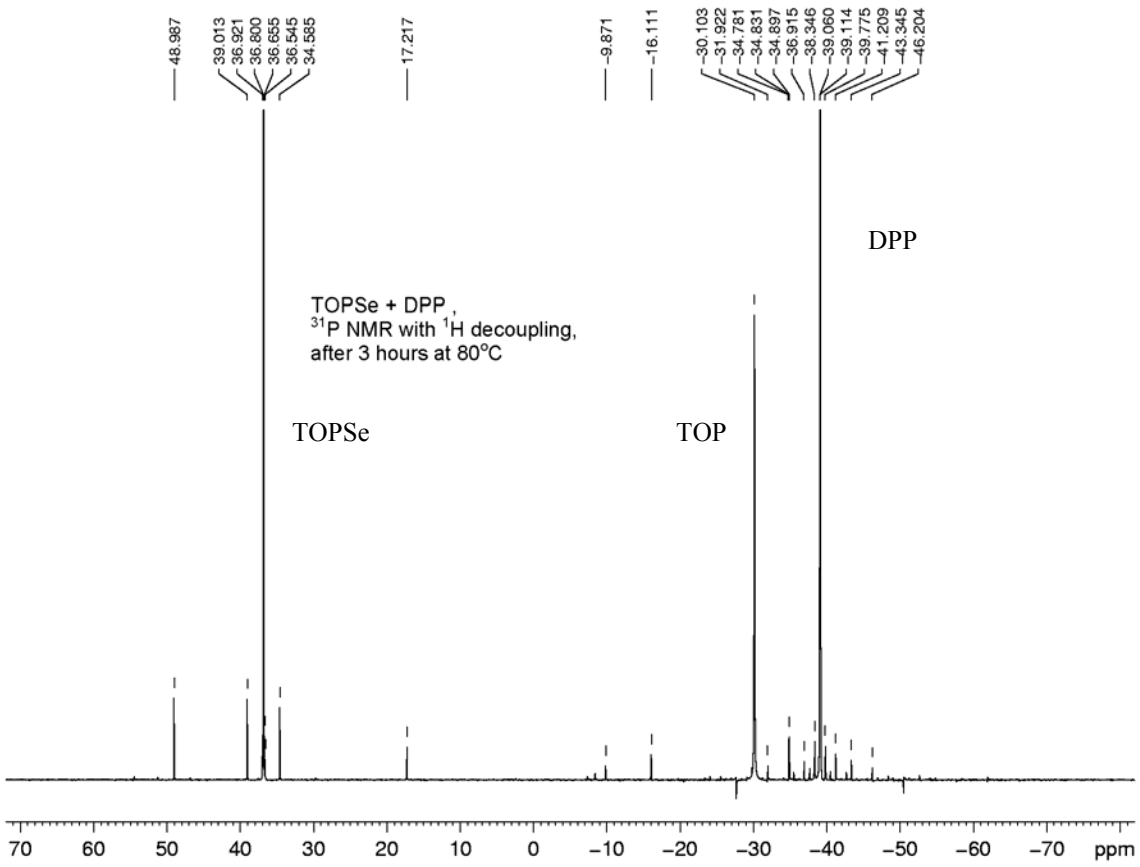


Scheme S1. ^{31}P NMR detected little DPPSe (δ 7.5 ppm) (1) with the addition of DPP to TOPSe. The NMR sample was preparation was in a glovebox, where 0.43 mL (0.44 mmol) of 1.02 M TOPSe stock solution was mixed with DPP 0.31 mL (1.75 mmol). The mixture was loaded in a NMR tube and properly sealed. The NMR measurements were done right away at 25 °C, and then 45 °C/10 min, 80 °C/10 min and 80 °C/3 hours. The spectrum was taken after 3 hours at 80 °C.

(Ref 1=31) Evans, C. M.; Evans, M. E.; Krauss, T. D. *J. Am. Chem. Soc.* **2010**, *132*, 10973–10975.

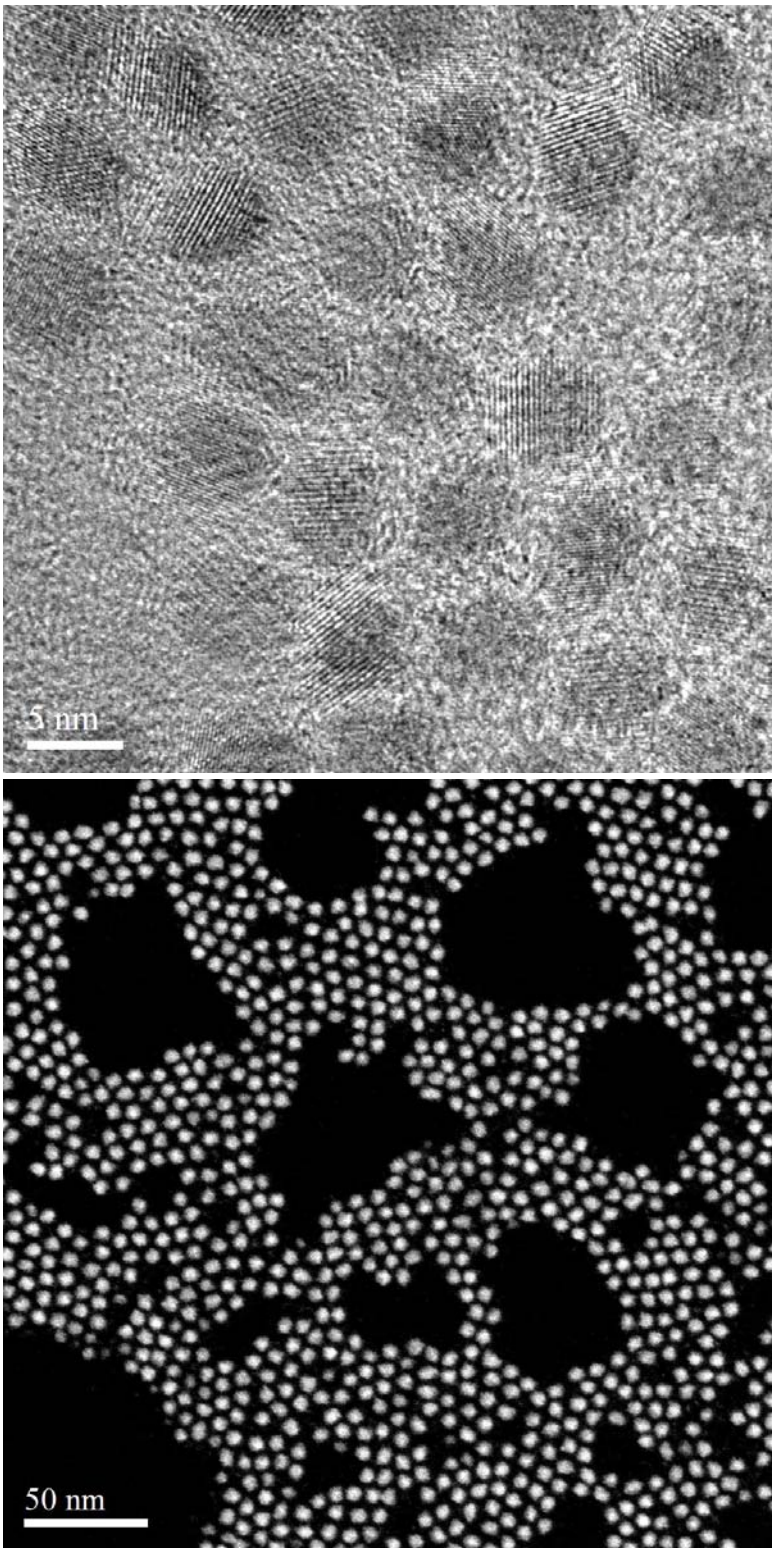


Figure S1A. A typical high resolution TEM image (top) and a HAADF-STEM image (bottom) of the PbSe NCs prepared from Batch b shown in Figure 1 (in the main text). The growth period was 25 minutes. The PbSe sample exhibited absorption peaking at 1528 nm. Based on the relationship between the absorption peak position and size (see ref 26 in the main text), the size is estimated to be 4.92 nm.

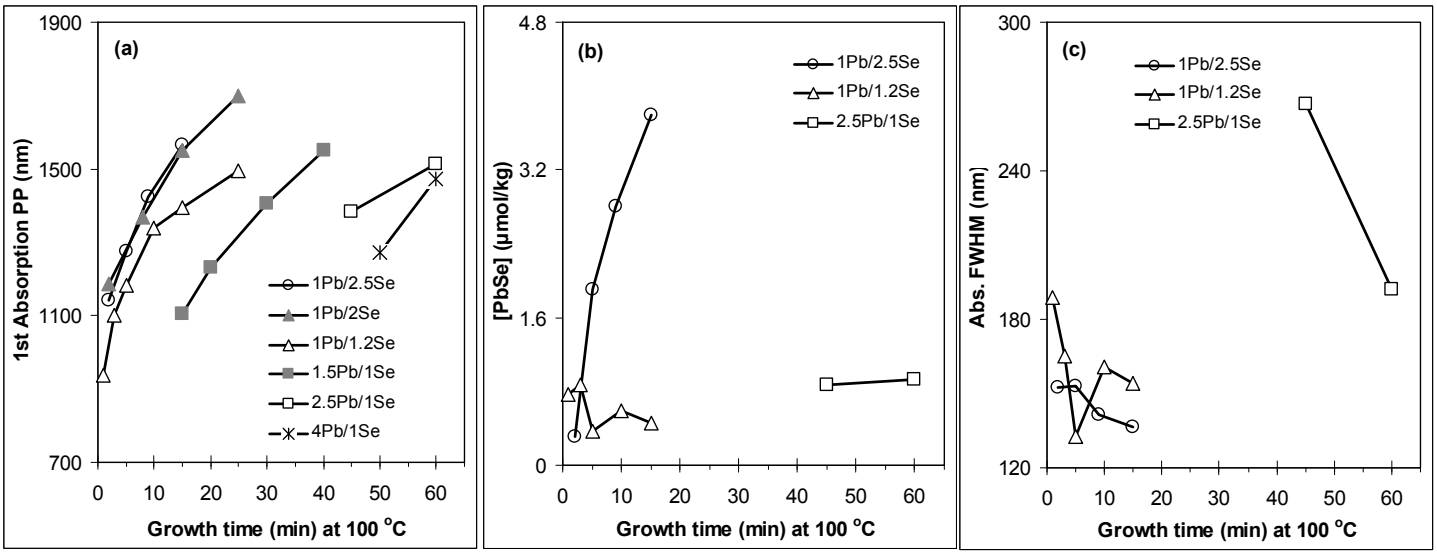


Figure S1B. Investigation on the effect of the Pb-to-TOPSe feed molar ratio affecting the formation of the PbSe NCs. (a) The first excitonic absorption peak position, (b) the [PbSe] in µmol/kg in the growth mixtures, and (c) absorption FWHM in nm of the resulting PbSe NCs from the batches studied. The Pb-to-TOPSe feed molar ratios of the batches are indicated, the [Pb] or [Se] (which was less) was 150 mmol/L. A total volume of the reaction medium was 8 mL scale, except 16 mL for Batch 1Pb-to-2.5Se. The growth temperature was 100 °C.

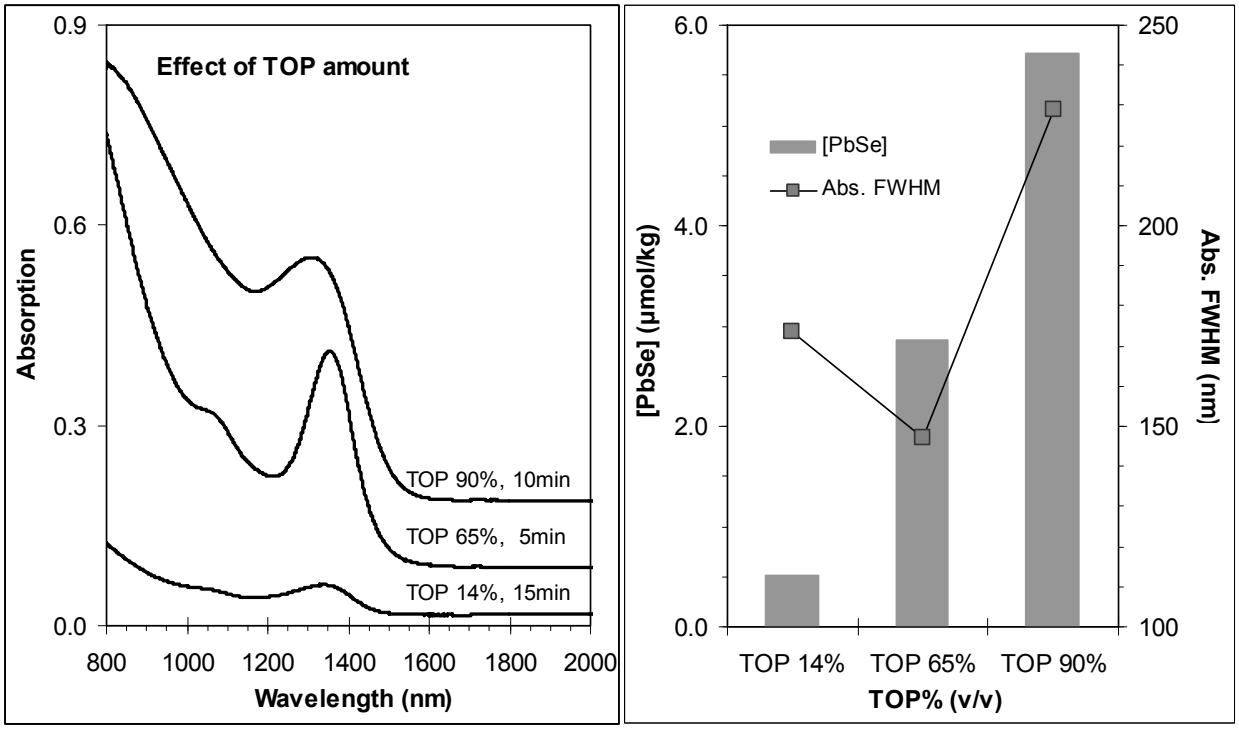


Figure S1C. Investigation on the effect of the TOP amount (v/v) used affecting the formation of PbSe NCs via our non-injection approach. (Left) The absorption (offset) of the PbSe NCs from the three batches with the feed molar ratio of 1.5Pb-to-1TOPSe and [Se] 150 mmol/L in ~8 mL reaction media. The amount of TOP in the reaction media is indicated, together with the growth periods for each of the three batches. The growth temperature was 100 °C. The absorption spectra were normalized to 1.0 gram of crude growth mixtures in 1.0 mL TCE. (Right) The [PbSe] in µmol/kg (left y axis) and absorption FWHM in nm of the three samples.

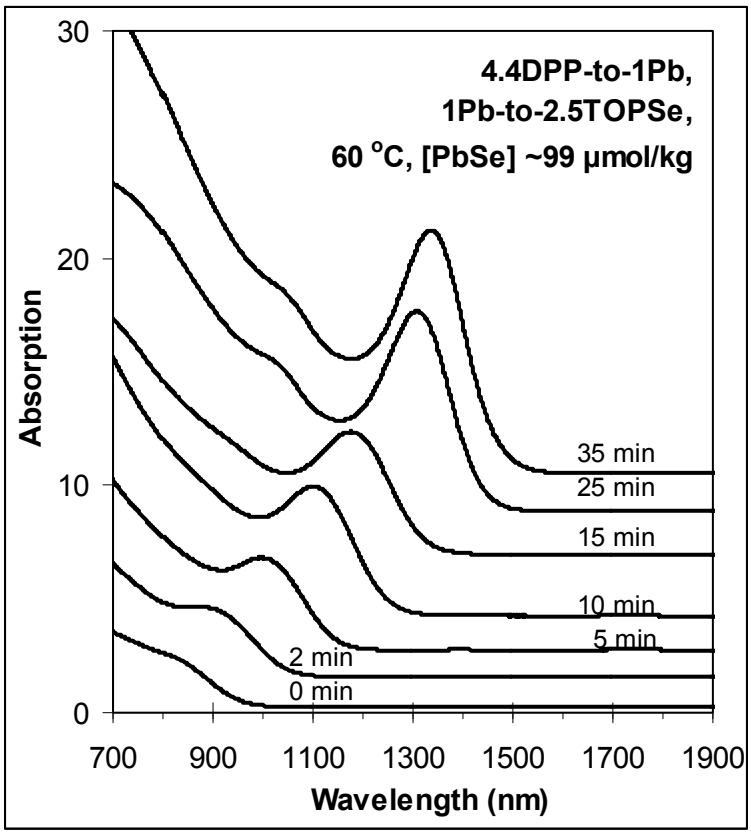


Figure S2. The temporal evolution of the absorption (offset) of the PbSe NCs synthesized with the feed molar ratio of 4.4DPP-to-1Pb and 1Pb-to-2.5TOPSe and [Pb] ~71 mmol/L in ~8 mL reaction medium. The growing temperature was 60 °C. The absorption spectra were normalized to 1.0 gram of crude reaction mixtures in 1.0 mL TCE. It seems that the nucleation/growth was faster than that of Batch b shown in Figure 2 in the main text.

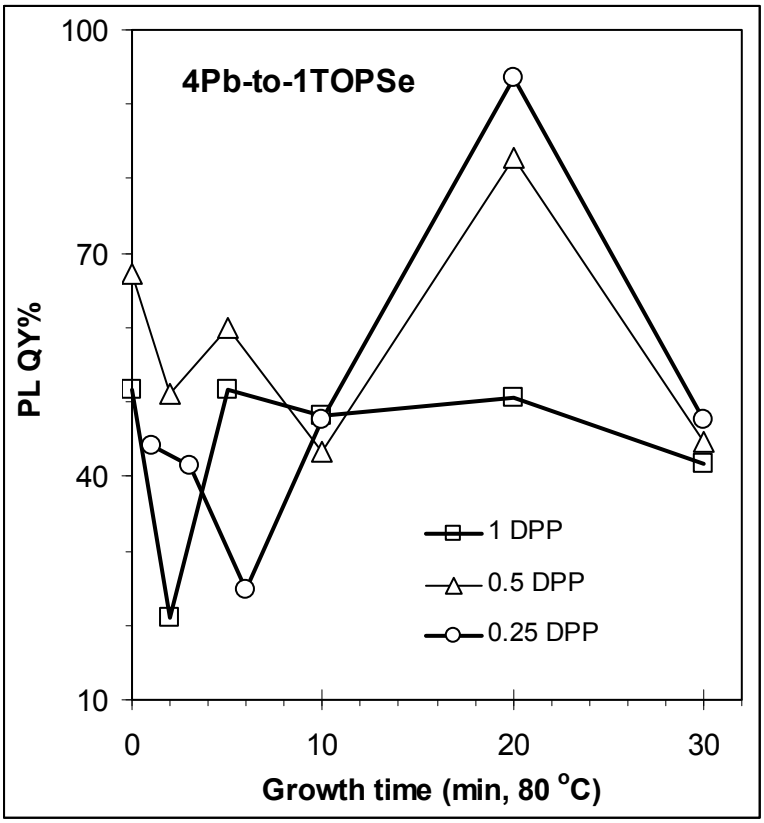


Figure S3A. The PL QY of the PbSe NCs synthesized from the batches shown in Figure 3.

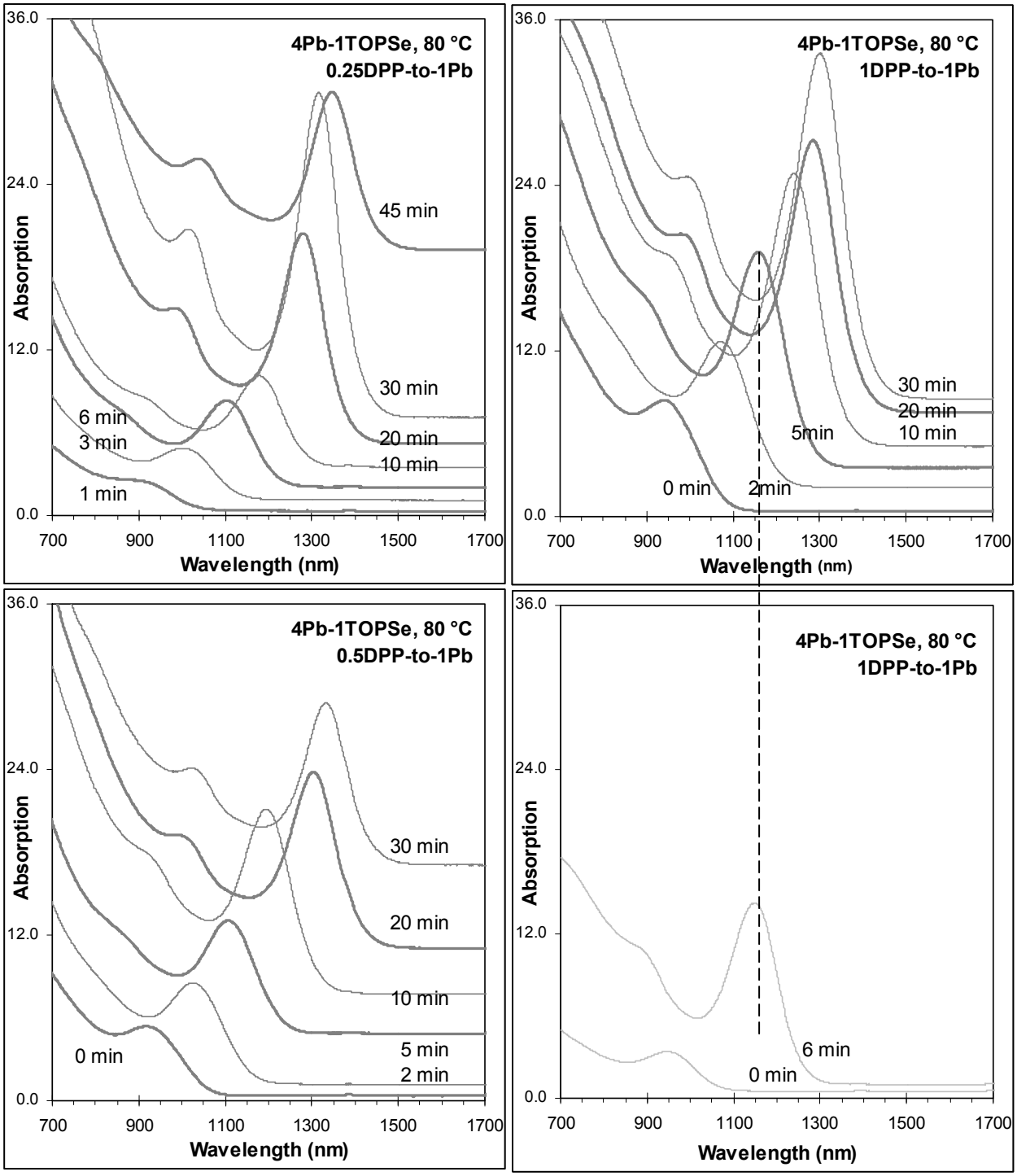
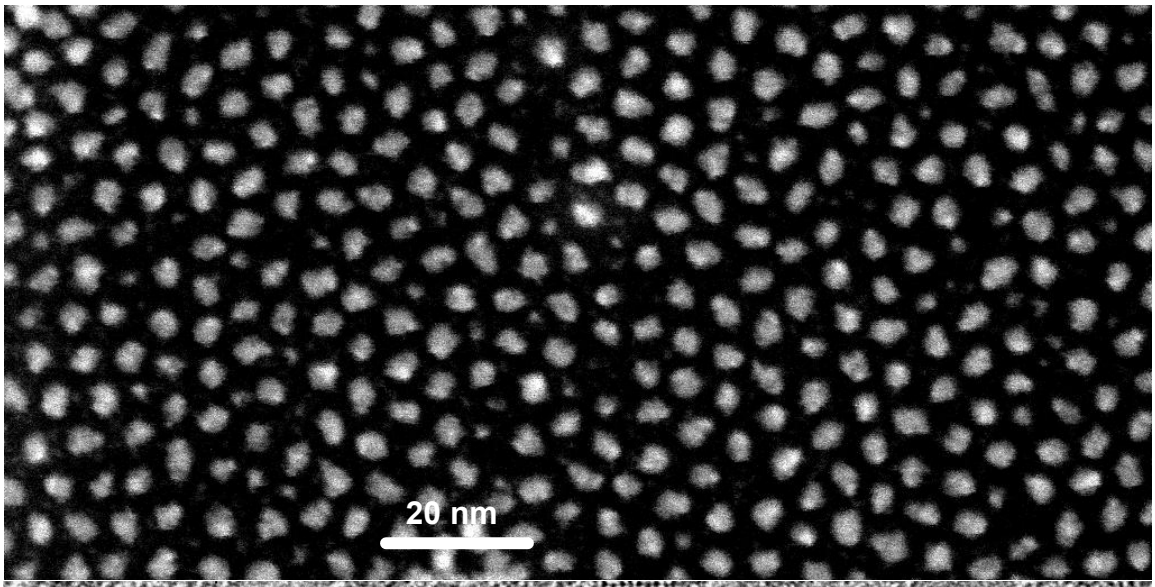


Figure S3B. Temporal evolution of the absorption (offset) of the PbSe NCs from the three batches shown in Figure 3. The absorption spectra were normalized to 1.0 gram of crude growth mixtures and dispersed in 1.0 mL TCE. (Note that the 6-min sample of Batch right-bottom was used for TEM shown in Figure S3C; Batch right-bottom and Bath right-top had the same reaction condition. The high synthetic reproducibility is worthy of notice.


20 nm

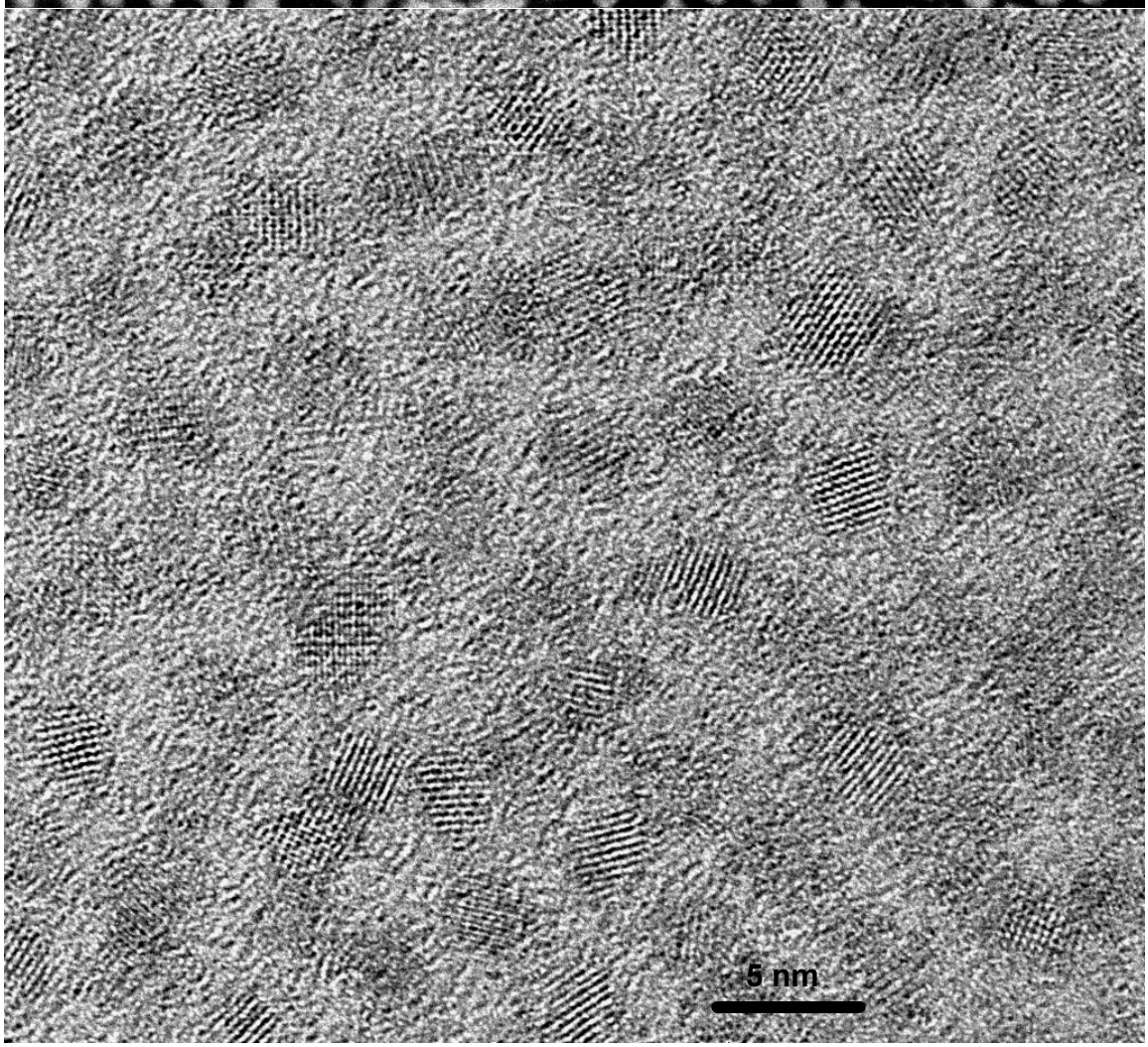

5 nm

Figure S3C. A typical HAADF-STEM (High Angle Annular Dark Field - Scanning Transmission Electron Microscope) image (top) and a high resolution TEM image (bottom) of our small-sized PbSe NCs. The PbSe NCs were prepared from Figure S3B Batch (right-bottom) which had the identical experimental condition as that shown in Figure 3 and Figure S3B Batch (right-top), with the feed molar ratio of 1DPP-to-1Pb and 4Pb-to-1TOPSe, and [Se] of ~ 71 mmol/kg; the growth temperature was 80 °C but the growth period was 6 min. The PbSe NCs exhibited their first excitonic absorption peaking at 1146 nm. The TEM mean size is ~3.6 nm with a standard deviation of ~9%. Based on the relationship between the absorption peak position and size (see ref 26 in the main text), the size is estimated to be 3.56 nm.

Table S1. For PL FWHM, both nm and meV were used by different groups. In order to compare with other peoples' data, such as Ref 5, and 30, we use the former. Here, the former is less dependent on peak position (namely NC size). For Samples 1 – 4 with absorption peak position redshifting from 1025.0 nm to 1246.5 nm, PL FWHM was calculated to be 150.1─156.4 nm, while 182.9 meV to 125.4 meV (which was monotonously decreased).

Fig 3 a and b	Growth time (min)	PL Peak Position (nm)	PL FWHM (nm)	PL FWHM (meV)
Sample 1	1	1025.0	154.0	182.9
Sample 2	3	1096.5	150.1	155.6
Sample 3	6	1176.5	154.1	138.8
Sample 4	10	1246.5	156.4	125.4
Sample 5	20	1332.1	140.1	98.2
Sample 6	30	1362.4	143.2	96.0

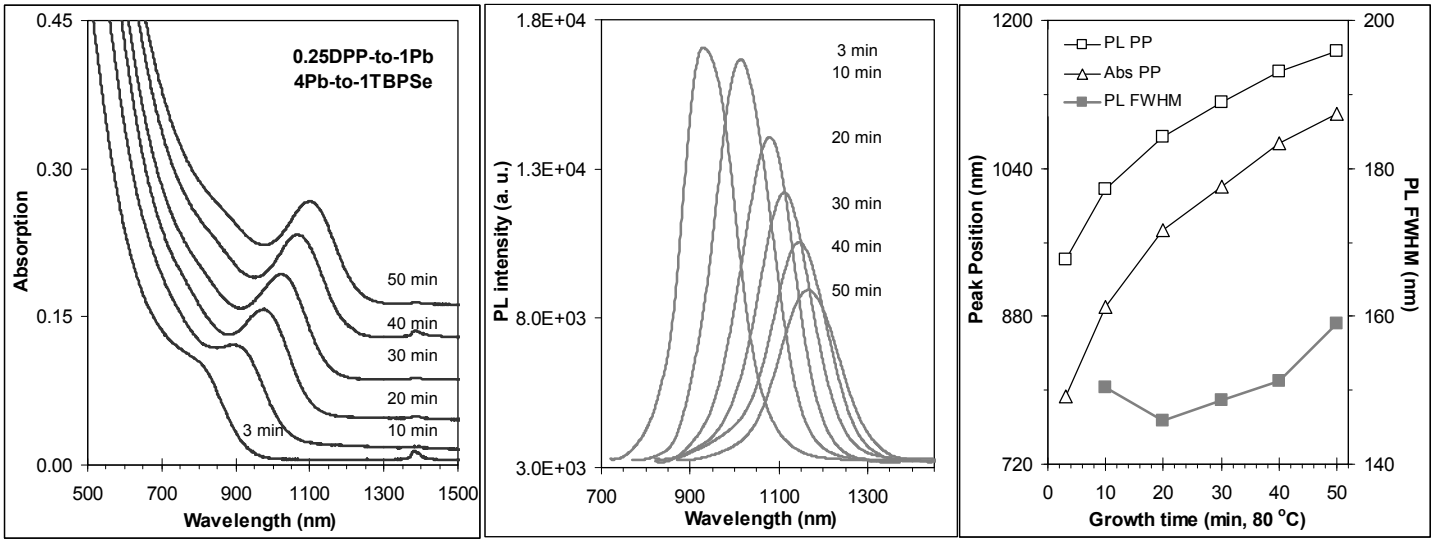


Figure S3D. The effect of the presence of DPP on the formation of PbSe NCs with the feed molar ratio of 4Pb(oleate)$_2$-to-1TBPSe, [Se] of ~71 mmol/kg, and growth temperature 80 °C within the growth periods of 50 min. Similarly, with the feed molar ratio of 4Pb(oleate)$_2$-to-1TBPSe, the use of DPP (Method (5)) could also help achieve balanced nucleation and growth, leading to small-sized PbSe NCs with high quality. The amount of DPP used matched the feed molar ratio of 0.25DPP-to-1Pb. For this batch, the TBPSe stock solution was prepared with the feed molar ratio of 1Se-to-5.9TBP instead of 1Se-to-2.2TBP. The resulting PbSe NCs exhibited the redshift of their absorption peak positions from 793 nm to 1099 nm, and the redshift of their emission peak positions from 942 nm to 1167 nm. The PL FWHM was ~146─151 nm in 10─40 min, followed by an increase to ~159 nm at 50 min. An averaged value of ~45 μmol/kg of the [PbSe] was obtained, while PL QY was relatively stable at 61─64% within 10 min growth and decreased continuously to 34 % afterwards. (Left) The temporal evolution of absorption (offset and normalized to the same height of the first excitonic peaks), (middle) the temporal evolution of emission (normalized to 0.1 optical density at the excitation wavelengths and corrected by IGA photodetector response sensitivity) of the PbSe NCs, and (right) the summary of the peak position (left y axis) of the bandgap absorption and emission in nm and PL FWHM in nm (right y axis).

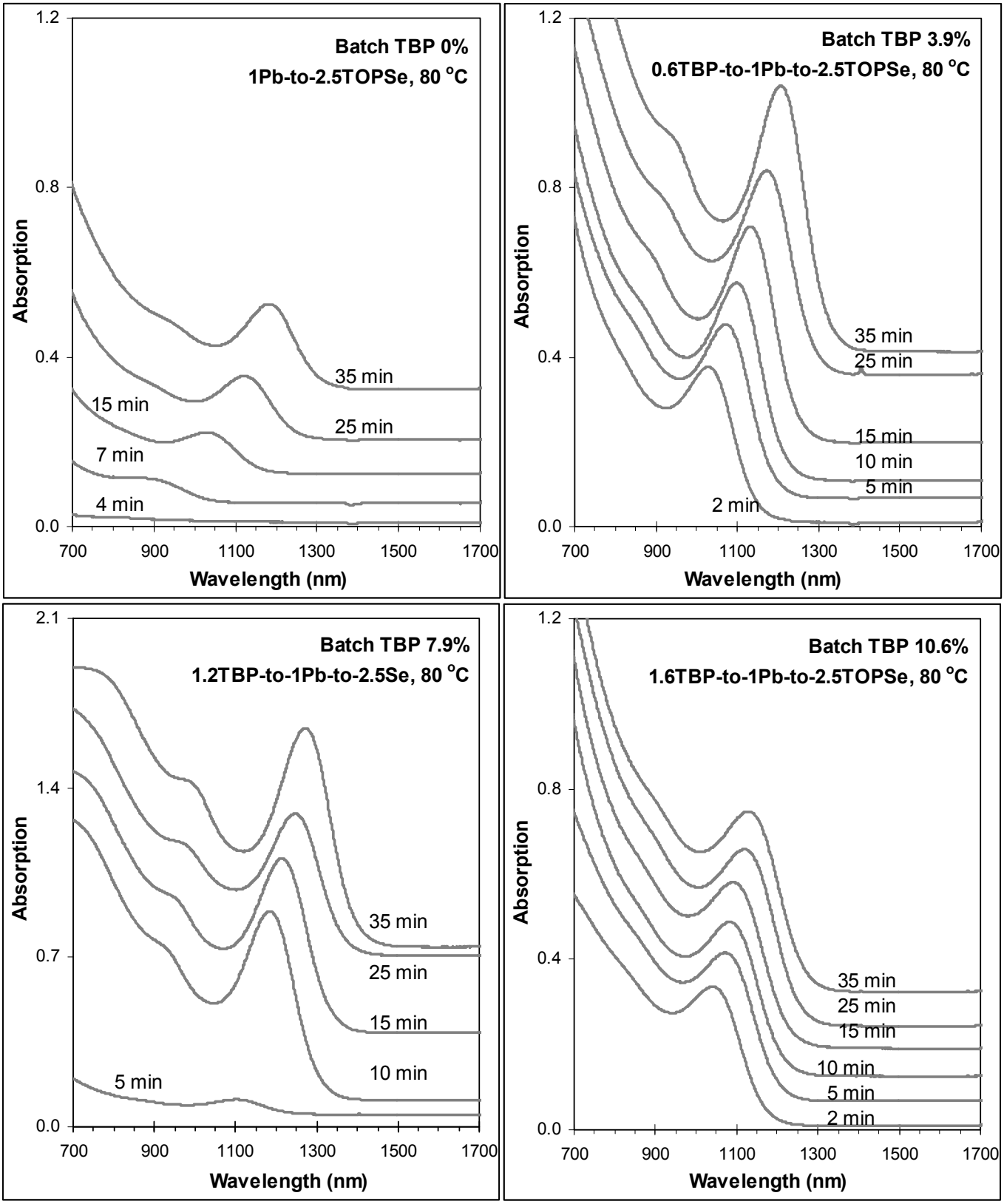
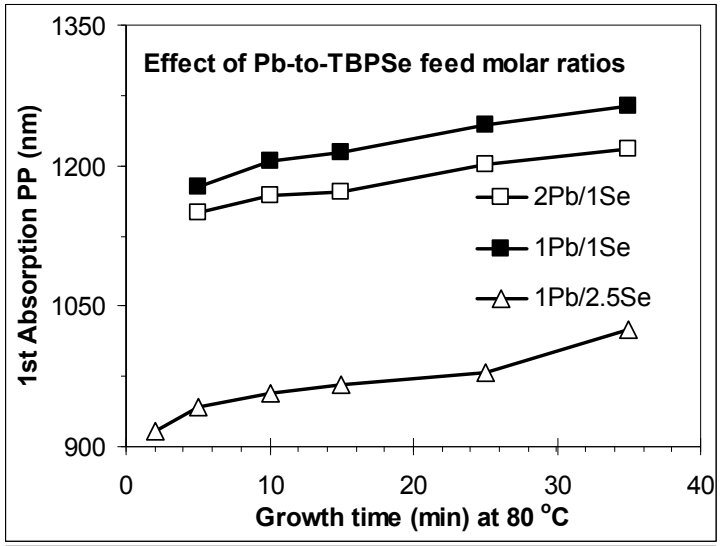
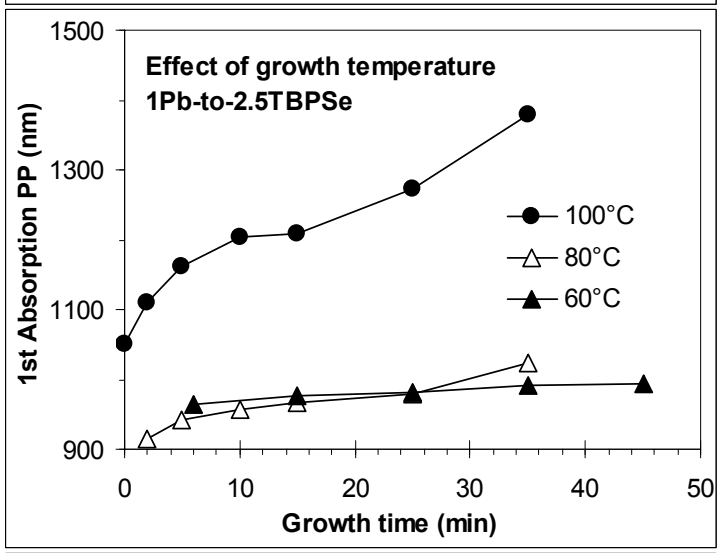

Batch TBP 0%
1Pb-to-2.5TOPSe, 80 °C
Absorption
Wavelength (nm)
35 min
25 min
15 min
7 min
4 min
Batch TBP 3.9%
0.6TBP-to-1Pb-to-2.5TOPSe, 80 °C
35 min
25 min
15 min
10 min
5 min
2 min
Batch TBP 7.9%
1.2TBP-to-1Pb-to-2.5Se, 80 °C
35 min
25 min
15 min
10 min
5 min
Batch TBP 10.6%
1.6TBP-to-1Pb-to-2.5TOPSe, 80 °C
35 min
25 min
15 min
10 min
5 min
2 min

Figure S4. Temporal evolution of the absorption (offset) of the PbSe NCs from the four batches shown in Figure 4. The absorption spectra of Batches TBP 0%, 3.9%, and 7.9% were normalized to 1.0 gram of crude growth mixtures and dispersed in 1.0 mL TCE. Here, Batches TBP 0%, 3.9%, 7.9%, and 10.6% are corresponding to the expression of Batches no TBP, 0.6TBP-to-1Pb, 1.2TBP-to-1Pb, and 1.6TBP-to-1Pb, respectively.


Effect of Pb-to-TBPSe feed molar ratios
1st Absorption PP (nm)
1350
1200
1050
900
0
10
20
30
40
Growth time (min) at 80 °C
2Pb/1Se
1Pb/1Se
1Pb/2.5Se


Effect of growth temperature
1Pb-to-2.5TBPSe
1st Absorption PP (nm)
1500
1300
1100
900
0
10
20
30
40
50
Growth time (min)
100°C
80°C
60°C

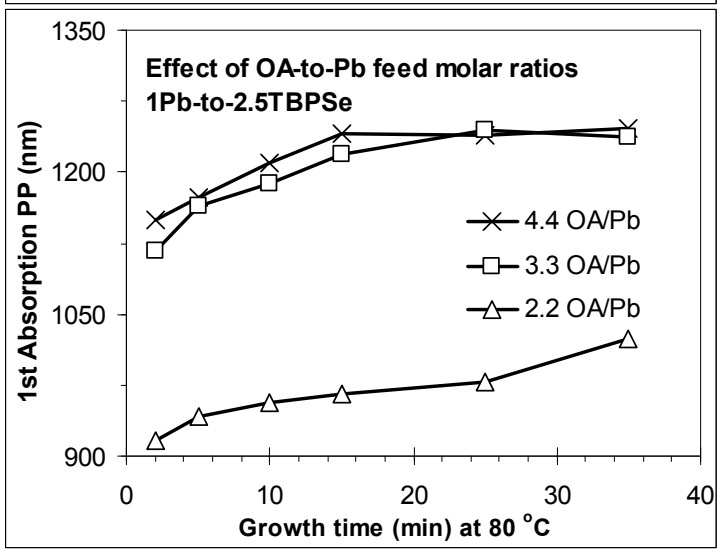

Effect of OA-to-Pb feed molar ratios
1Pb-to-2.5TBPSe
1st Absorption PP (nm)
1350
1200
1050
900
0
10
20
30
40
Growth time (min) at 80 °C
4.4 OA/Pb
3.3 OA/Pb
2.2 OA/Pb

Figure S5. Investigation on the synthetic parameters affecting the formation of PbSe NCs when TBPSe was used as the Se precursor. Here, the [Pb] or [Se] (which was less) was ~71 mmol/L. The parameters studied include the feed molar ratio of Pb-to-Se (top, with the growth temperature of 80 °C and the feed molar ratio of 2.2OA-to-1Pb), the growth temperature (middle, with the feed molar ratio of 2.2OA-to-1Pb and 1Pb-to-2.5Se), and the feed molar ratio of OA-to-Pb (bottom, with the feed molar ratio of 1Pb-to-2.5Se and the growth temperature of 80 °C). The summary on the resulting [PbSe] in μmol/kg and absorption FWHM in nm, is shown in Supporting Information Figure S5A, together with the temporal evolution of the absorption of the PbSe NCs from the various batches studied in Supporting Information Figure S5B.

For the two batches shown in Figure 5, it is necessary to point out that, both the TOPSe and TBPSe stock solutions were prepared with the feed molar ratio of 1Se-to-2.2TOP and 1Se-to-2.2TBP, respectively. However, there were 18% TOP (v/v) and 10% TBP (v/v) of the 8 mL reaction media shown in Figure 5, respectively. The TBP amount used in Figure 5 Batch b is almost identical to that of Figure 4 Batch 1.6TBP-to-1Pb (with 10.6% TBP added); however, Table 1 suggests that Batch 1.6TBP-to-1Pb of Figure 4 was less powerful than Batch b of Figure 5, regarding the formation of the monomer leading to sizeable nucleation at 80 °C. Therefore, it seems reasonable that DBP/TBP added in Figure 4 acted as reducing agents, enhancing Route b (Equation (bM)); DBPSe/TBPSe in Figure 5 enhanced Route a (Equation (aM).

To better understand the nucleation/growth (Method (4)), a systematic investigation on the experimental parameters, including the Pb-to-TBPSe feed molar ratio, growth temperature, and ligand (oleic acid, OA) concentration, was performed and summarized in Figure S5, S5A, and S5B. It is interesting that for the three batches shown in Figure S5 (top) with the feed molar ratio of 2.2OA-to-1Pb and the growth at 80 °C, the resulting PbSe NCs exhibited an increase in size from Batch 1Pb-to-2.5Se, to 2Pb-to-1Se, and to 1Pb-to-1Se. The [Se] concentration was ~178 mmol/L, ~71 mmol/L, and ~71 mmol/L for the three batches, respectively. The difference between Batch 2Pb-to-1Se and Batch 1Pb-to-1Se may be related to the different diffusion capabilities. As mentioned in the main text, a high $Pb(oleate)_2$ concentration may cause the difficulty in diffusion in a reaction medium. Meanwhile, the difference between these two batches and Batch 1Pb-to-2.5Se may be due to the fact that the presence of [TBPSe] is associated with the presence of DBPSe and TBP. A high [TBPSe] led to a high [DBPSe] and [TBP]; DBPSe promoted Route a and thus sizeable nucleation.

Therefore, various synthetic parameters and the interplay between them affect the formation of the monomer and thus nucleation and growth. The optimal conditions with TBPSe as the precursor for PbSe NCs of various sizes are different. For PbSe NCs exhibiting bandgap absorption in the range of 1200─1300 nm, the 2Pb-to-1Se feed molar ratio, 60─80 °C growth temperature, and 3.3OA-to-1Pb feed molar ratio could be considered, to achieve narrow size distribution and high PL QY but with low particle yield. Meanwhile, for small-sized PbSe NCs with high yield and high quality, the 1Pb-to-(2─2.5)Se feed molar ratio, 60─80 °C growth temperature, and 2.2OA-to-1Pb feed molar ratio should be used. In general, a high Pb-to-Se ratio, high growth temperature, and a high OA-to-Pb ratio result in large NCs with a low NC yield. The low NC yield may be caused by the diffusion difficulty (under the condition of a high Pb-to-Se ratio), a decreased monomer saturation level (at high temperature), and a retarded precursor reactivity (with a high OA concentration) (2, 3). A low Pb-to-Se ratio, low growth temperature, and a low OA-to-Pb ratio lead to PbSe NCs with a high NC yield. This investigation on the synthetic parameters should shed some light into the design of the synthetic parameters for an essential balance between nucleation and growth.

(Ref 2=33) Ouyang, J.; Kuijper, J.; Brot, S.; Kingston, D.; Wu, X.; Leek, D. M.; Hu, M. Z.; Yu, K. *J. Phys. Chem. C* **2009**, *113*, 7579−7593.

(Ref 3=39) Yu, W. W.; Peng, X. *Angew. Chem. Int. Ed.* **2002**, *41*, 2368−2371.

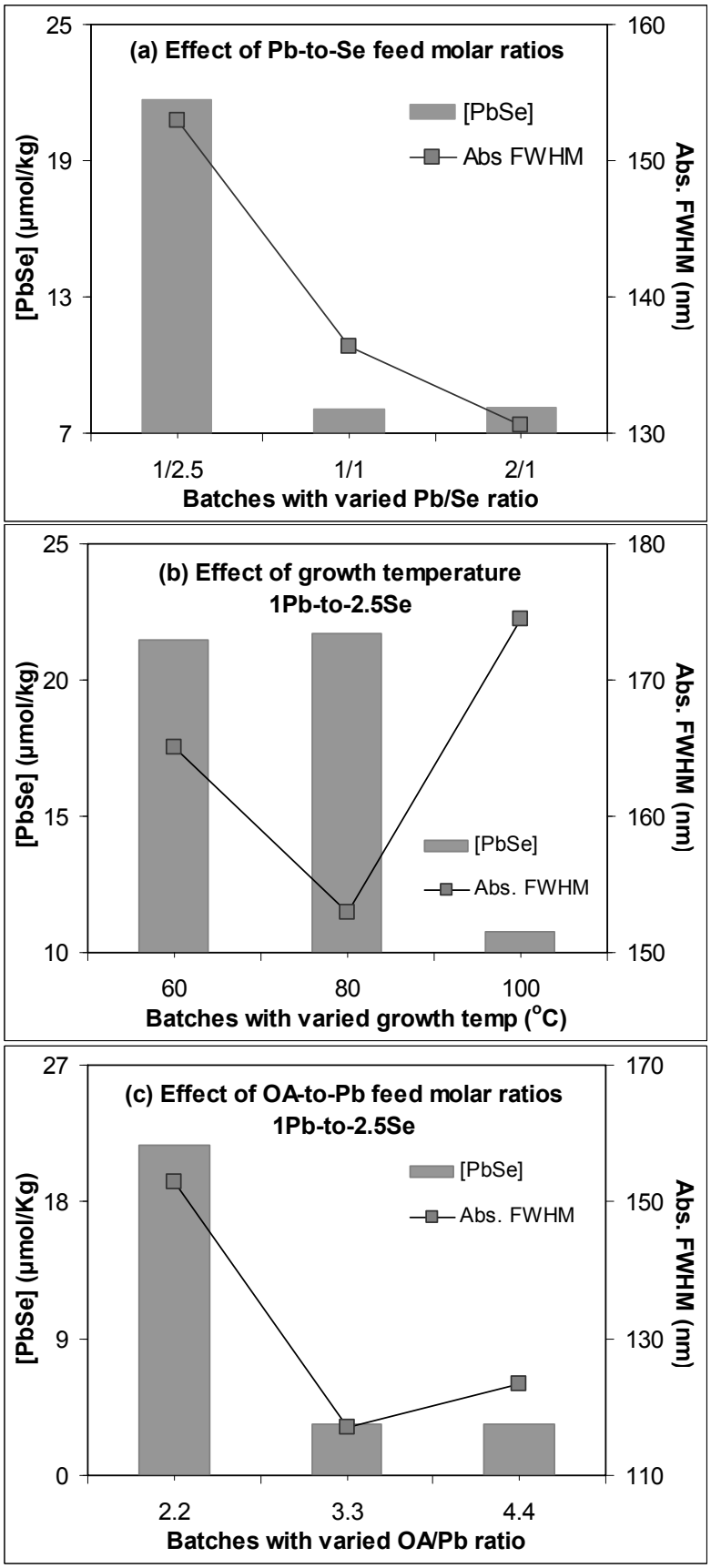
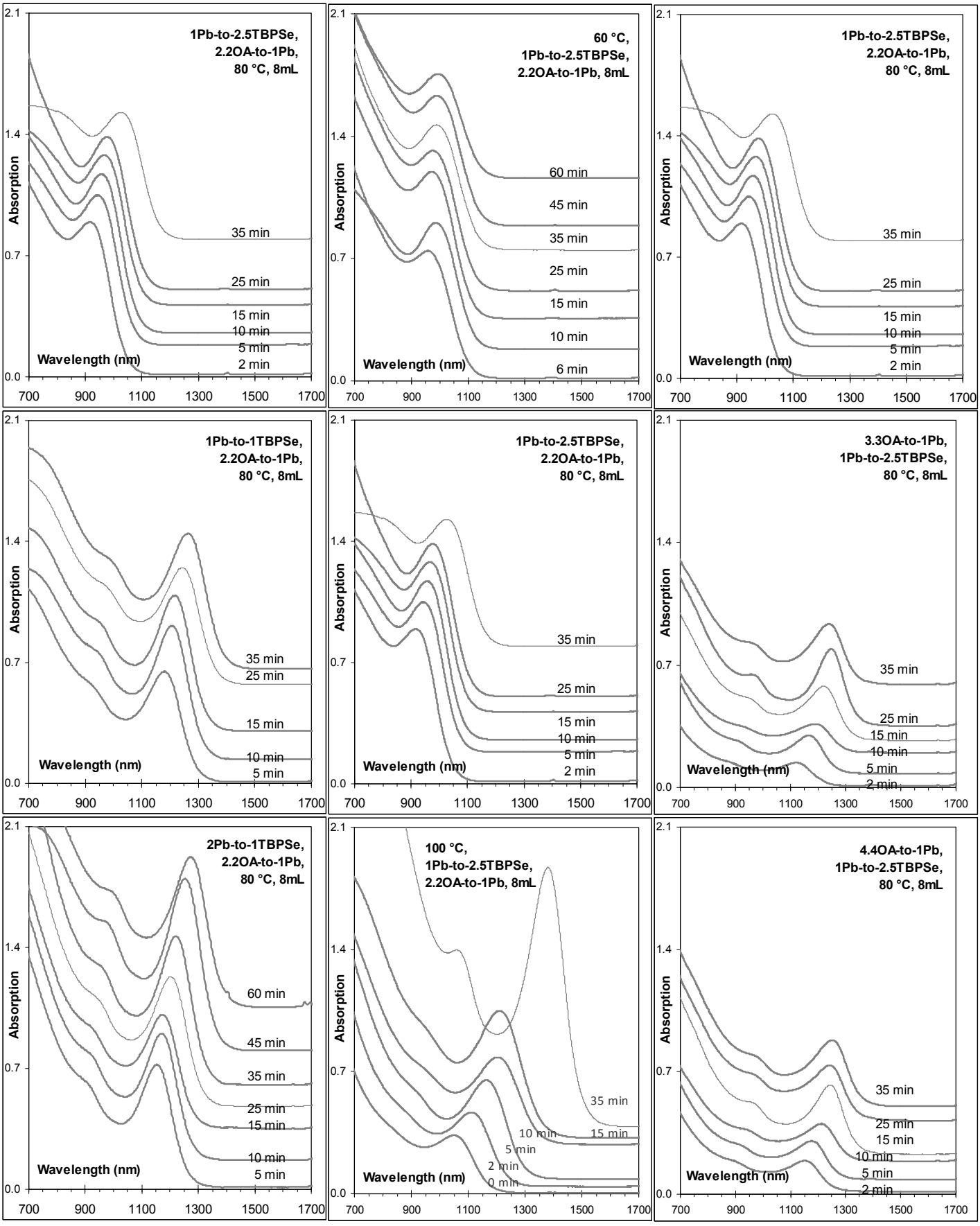
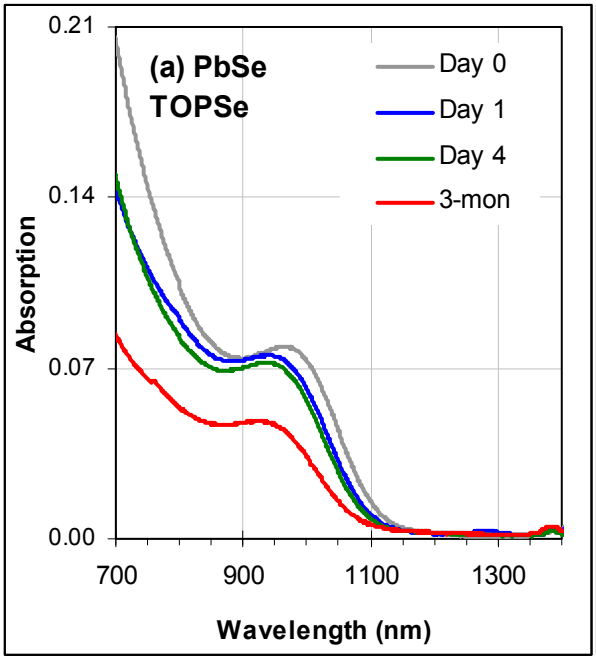
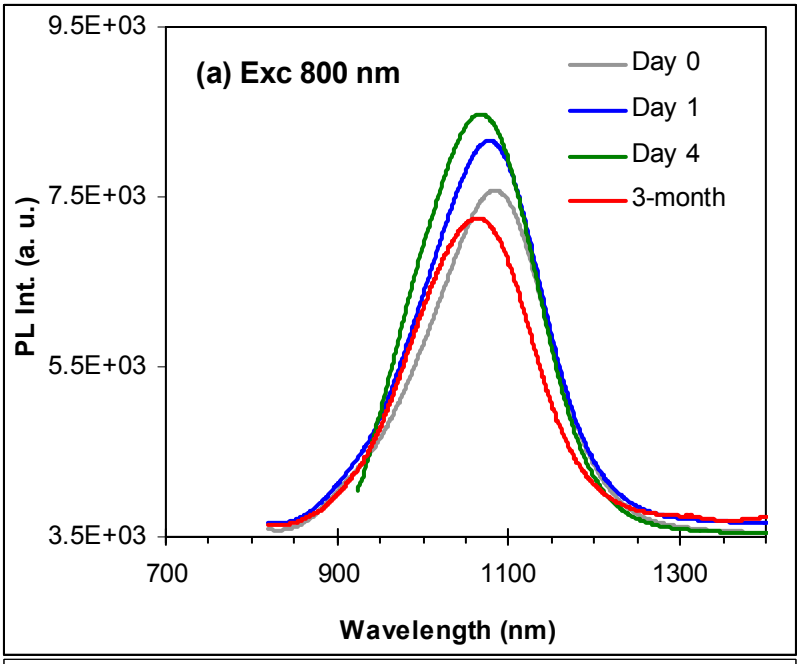

(a) Effect of Pb-to-Se feed molar ratios
[PbSe]
Abs FWHM
[PbSe] (µmol/kg)
Abs. FWHM (nm)
25
19
13
7
160
150
140
130
1/2.5
1/1
2/1
Batches with varied Pb/Se ratio
(b) Effect of growth temperature
1Pb-to-2.5Se
[PbSe] (µmol/kg)
Abs. FWHM (nm)
25
20
15
10
180
170
160
150
[PbSe]
Abs. FWHM
60
80
100
Batches with varied growth temp (°C)
(c) Effect of OA-to-Pb feed molar ratios
1Pb-to-2.5Se
[PbSe] (µmol/Kg)
Abs. FWHM (nm)
27
18
9
0
170
150
130
110
[PbSe]
Abs. FWHM
2.2
3.3
4.4
Batches with varied OA/Pb ratio

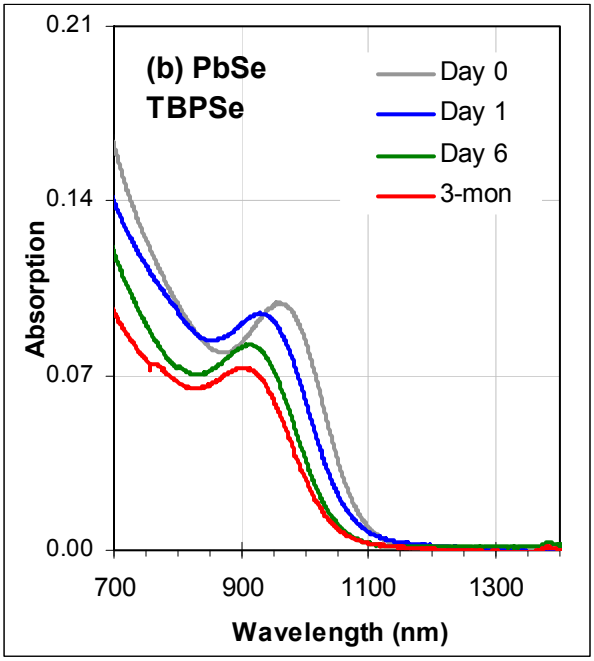
Figure S5A. For the PbSe NCs shown in Figure S5 (top), (middle), and (bottom), the [PbSe] in μmol/kg (left y axis) and absorption FWHM in nm (right y axis) are summarized in (a), (b), and (c), respectively. The data were averaged from those obtained from the 15- and 25-min samples of each batch.

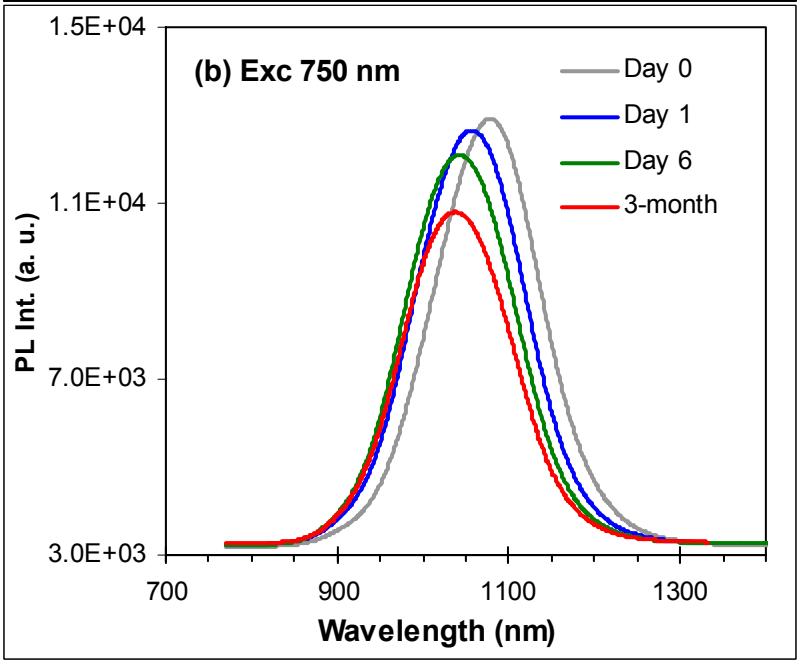
Figure S5B. Temporal evolution of the absorption (offset) of the PbSe NCs from the seven batches shown in Figure S5. The absorption spectra were normalized to 1.0 gram of crude growth mixtures and dispersed in 1.0 mL TCE. The three batches shown in the Left panel are those (a) to study the effect of the Pb-to-TBPSe feed molar ratio, while those (b) in the Middle panel are for the effect of the growth temperature, and those (c) in the Right panel are for the effect of OA-to-Pb feed molar ratios.

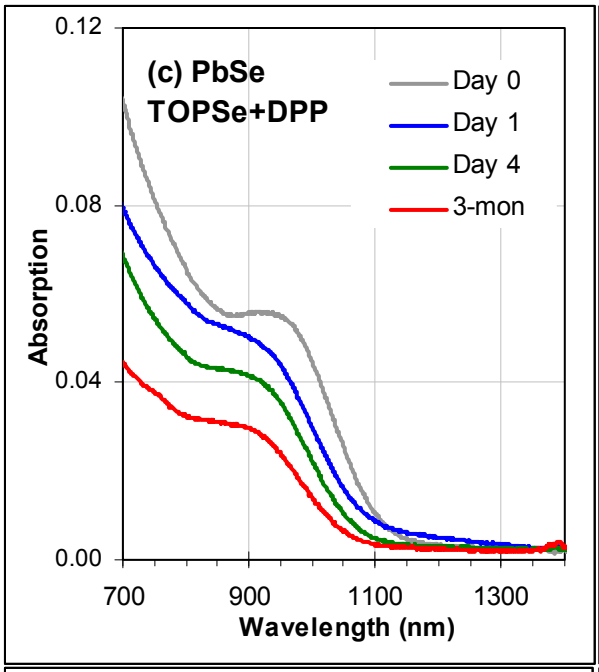

1Pb-to-2.5TBPSe,
2.2OA-to-1Pb,
80 °C, 8mL
35 min
25 min
15 min
10 min
5 min
2 min
60 °C,
1Pb-to-2.5TBPSe,
2.2OA-to-1Pb, 8mL
60 min
45 min
35 min
25 min
15 min
10 min
6 min
1Pb-to-1TBPSe,
2.2OA-to-1Pb,
80 °C, 8mL
3.3OA-to-1Pb,
1Pb-to-2.5TBPSe,
80 °C, 8mL
2Pb-to-1TBPSe,
2.2OA-to-1Pb,
80 °C, 8mL
100 °C,
1Pb-to-2.5TBPSe,
2.2OA-to-1Pb, 8mL
0 min
4.4OA-to-1Pb,
1Pb-to-2.5TBPSe,
80 °C, 8mL
Absorption
Wavelength (nm)
0.0
0.7
1.4
2.1
700
900
1100
1300
1500
1700

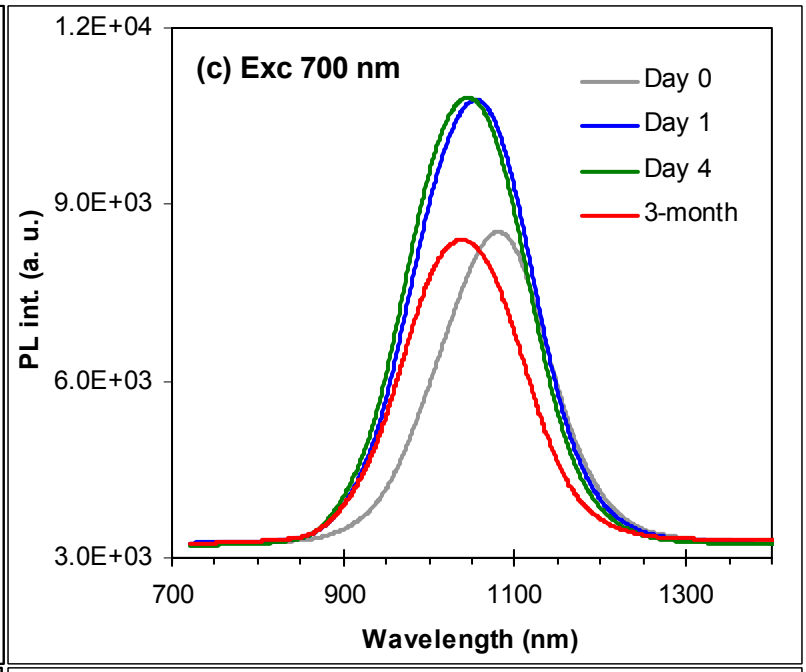

(a) PbSe
TOPSe
Day 0
Day 1
Day 4
3-mon
Absorption
0.21
0.14
0.07
0.00
700
900
1100
1300
Wavelength (nm)

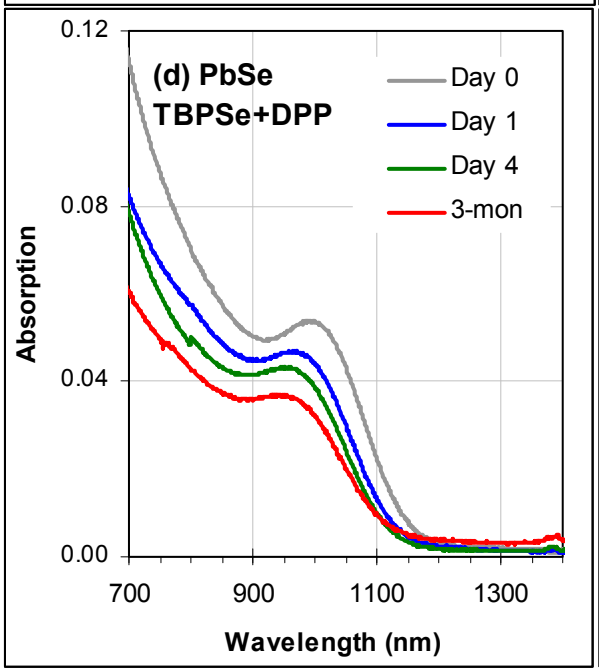

(a) Exc 800 nm
Day 0
Day 1
Day 4
3-month
PL Int. (a. u.)
9.5E+03
7.5E+03
5.5E+03
3.5E+03
700
900
1100
1300
Wavelength (nm)


(b) PbSe
TBPSe
Day 0
Day 1
Day 6
3-mon
Absorption
0.21
0.14
0.07
0.00
700
900
1100
1300
Wavelength (nm)


(b) Exc 750 nm
Day 0
Day 1
Day 6
3-month
PL Int. (a. u.)
1.5E+04
1.1E+04
7.0E+03
3.0E+03
700
900
1100
1300
Wavelength (nm)


(c) PbSe
TOPSe+DPP
Day 0
Day 1
Day 4
3-mon
Absorption
0.12
0.08
0.04
0.00
700
900
1100
1300
Wavelength (nm)


(c) Exc 700 nm
Day 0
Day 1
Day 4
3-month
PL int. (a. u.)
1.2E+04
9.0E+03
6.0E+03
3.0E+03
700
900
1100
1300
Wavelength (nm)


(d) PbSe
TBPSe+DPP
Day 0
Day 1
Day 4
3-mon
Absorption
0.12
0.08
0.04
0.00
700
900
1100
1300
Wavelength (nm)

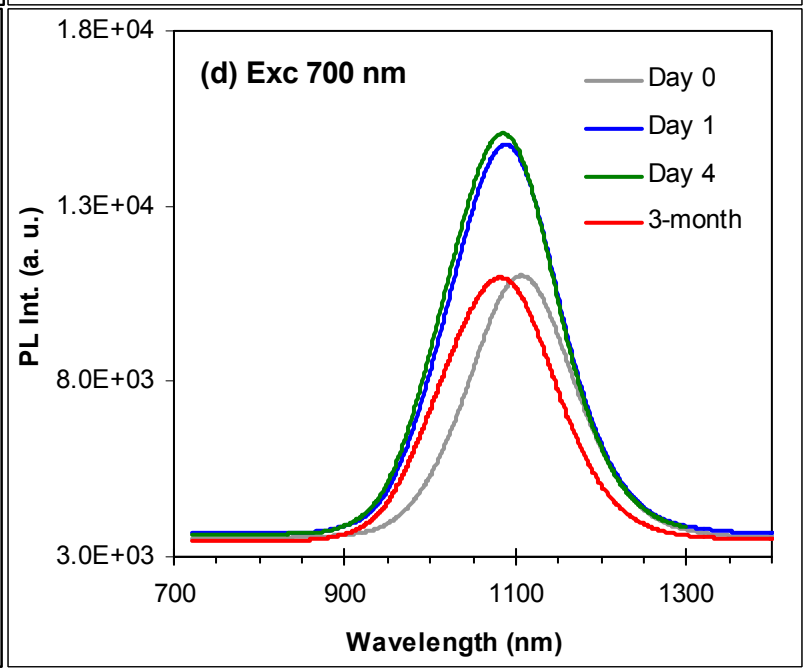

(d) Exc 700 nm
Day 0
Day 1
Day 4
3-month
PL Int. (a. u.)
1.8E+04
1.3E+04
8.0E+03
3.0E+03
700
900
1100
1300
Wavelength (nm)

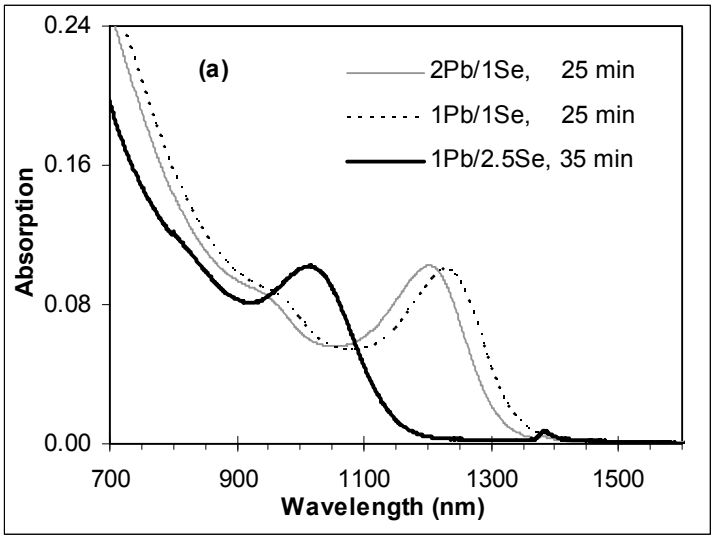
Figure S6A. Investigation on the change of the absorption (left) and emission (right), during dark storage at -20 °C with air contact up to three months. Our small-sized PbSe NCs exhibited excellent storage stability. The solvent used to disperse the PbSe NCs was TCE. Here, similar-sized PbSe NCs exhibiting bandgap emission peaking at ~1100 nm were chosen. These PbSe NCs were synthesized from our four synthetic batches, representing the various methods developed with our non-injection-based synthetic protocol targeting small-sized PbSe NCs with high quality and high reaction yield. The experimental conditions are, with [Pb] ~71 mmol/L,

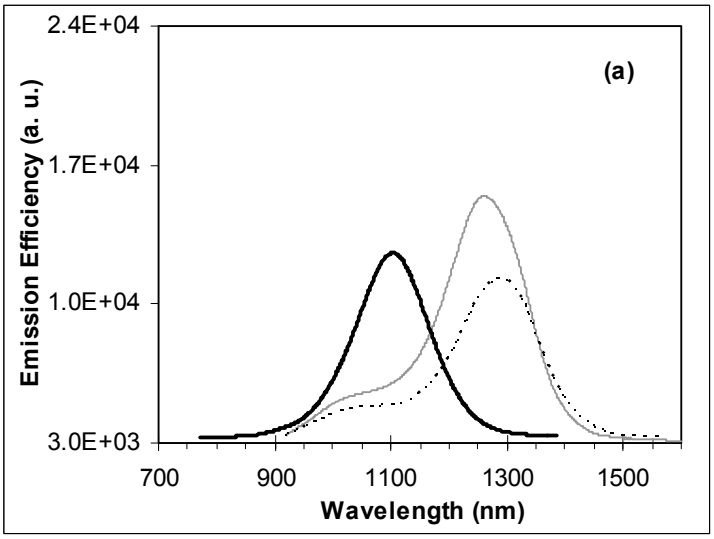
Batch a: 1Pb-to-2.5TOPSe, 80 °C growth temperature for 10 min. (Method (1)).

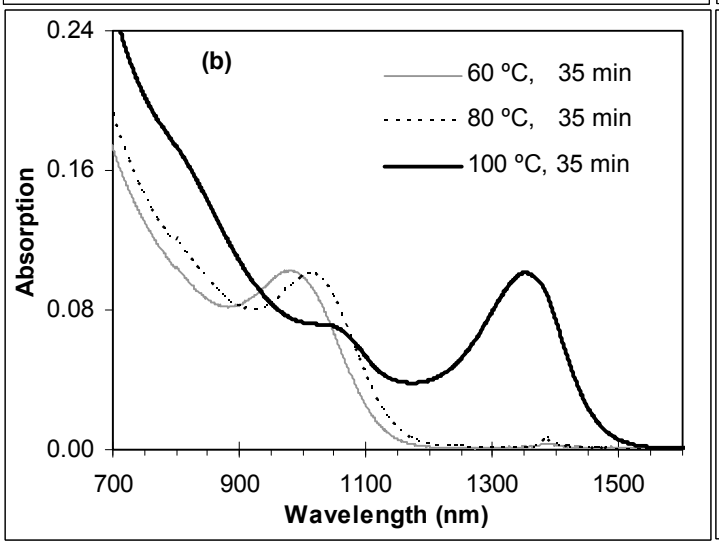
Batch b: 1Pb-to-2.5TBPSe, 80 °C growth temperature for 15 min. (Method (4)).

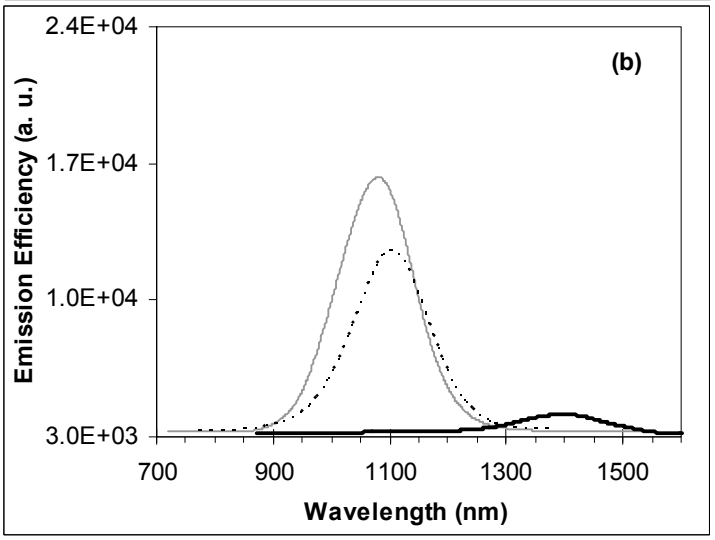
Batch c: 1.47DPP-to-1Pb-to-2.5TOPSe, 50 °C growth temperature for 15 min. (Method (2)).

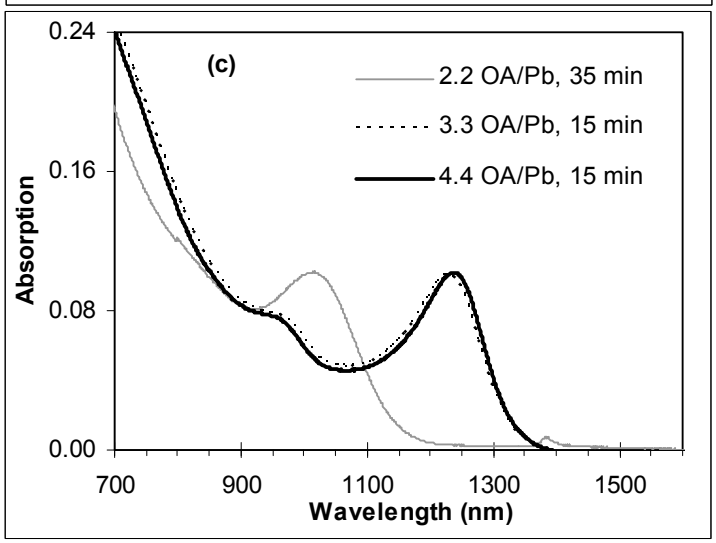
Batch d: 1.47DPP-to-1Pb-to-2.5TBPSe, 50 °C growth temperature for 15 min. (Method (5)).


(a)
2Pb/1Se, 25 min
1Pb/1Se, 25 min
1Pb/2.5Se, 35 min
Absorption
Wavelength (nm)
0.24
0.16
0.08
0.00
700
900
1100
1300
1500


(a)
Emission Efficiency (a. u.)
Wavelength (nm)
2.4E+04
1.7E+04
1.0E+04
3.0E+03
700
900
1100
1300
1500


(b)
60 °C, 35 min
80 °C, 35 min
100 °C, 35 min
Absorption
Wavelength (nm)
0.24
0.16
0.08
0.00
700
900
1100
1300
1500


(b)
Emission Efficiency (a. u.)
Wavelength (nm)
2.4E+04
1.7E+04
1.0E+04
3.0E+03
700
900
1100
1300
1500


(c)
2.2 OA/Pb, 35 min
3.3 OA/Pb, 15 min
4.4 OA/Pb, 15 min
Absorption
Wavelength (nm)
0.24
0.16
0.08
0.00
700
900
1100
1300
1500

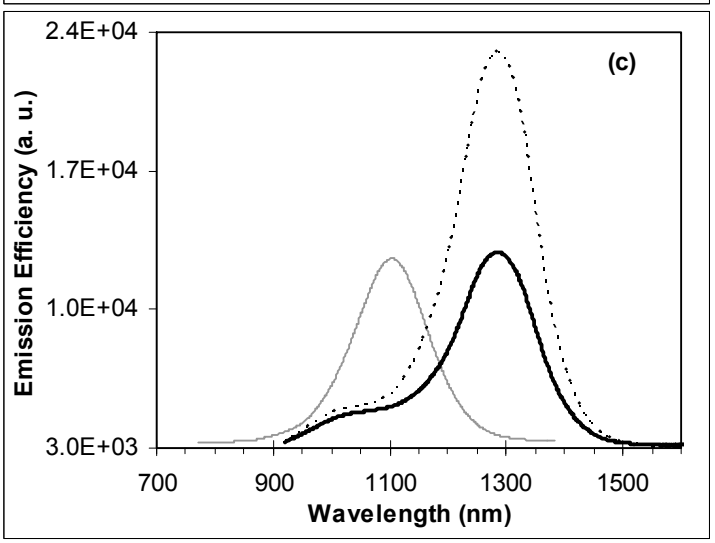

(c)
Emission Efficiency (a. u.)
Wavelength (nm)
2.4E+04
1.7E+04
1.0E+04
3.0E+03
700
900
1100
1300
1500

Figure S6B. Investigation on the stability of the PbSe NCs prepared from the batches shown in Figures S5, S5A, and S5B. After synthesized, the PbSe samples were purified and dispersed in TCE. Such concentrated TCE dispersions were stored at -20 ^{o}C for ~3.5 months. Then, the samples were diluted with TCE and the optical measurements were performed. (Left panel) Absorption (normalized to the same height of the excitonic absorption peaks) and (right panel) emission (normalized to 0.1 optical density at the exitation wavelengths and corrected with IGA detector response sensitivity). The change of the absorption peak position before and after storage is summarized in **Table S2**, together with the PL QY after the storage. The excellent stability is worthy of notice.

Table S2. The change of the absorption peak position of the PbSe NCs shown in Figures S5, S5A, and S5B, before and after storage. Here, the blueshift is negative and the redshift is positive.

	Batches	Growth period/Temp	Abs PP, as synthesized	Abs PP, after ~3.5 months	Abs PP Shift	PL QY after ~3.5 months
	1Pb-to-2.5Se	35min/80 ºC	1024 nm	1013 nm	-11 nm	44%
(a)	1Pb-to-1Se	25min/80 ºC	1243 nm	1225 nm	-18 nm	36%
Pb/Se effect	2Pb-to-1Se	25min/80 ºC	1201 nm	1198 nm	-3 nm	59%
(b)	60 ºC	35 min	992 nm	982 nm	-10 nm	64%
Temperature effect	80 ºC	35 min	1024 nm	1013 nm	-11 nm	44%
	100 ºC	35 min	1378 nm	1351 nm	-27 nm	5%
(c)	2.2OA-to-1Pb	35 min/80 ºC	1024 nm	1013 nm	-11 nm	44%
OA/Pb effect	3.3OA-to-1Pb	15 min/80 ºC	1218 nm	1226 nm	+ 8 nm	98%
	4.4OA-to-1Pb	15 min/80 ºC	1239 nm	1234 nm	- 5 nm	46%

Here, the NCs with high QYs exhibited good storage stability. Usually, high QY indicates good surface passivation with few defects. Optimized synthetic methods with optimized experimental conditions lead to balanced nucleation/growth, with the resulting NCs having few defects while high QYs and good storage stability. The use of DBPSe/TBPSe, DBP/TBP, or DPP led to balanced nucleation/growth together with optimized synthetic conditions such as Pb-to-Se and OA-to-Pb feed molar ratios, and temperature.

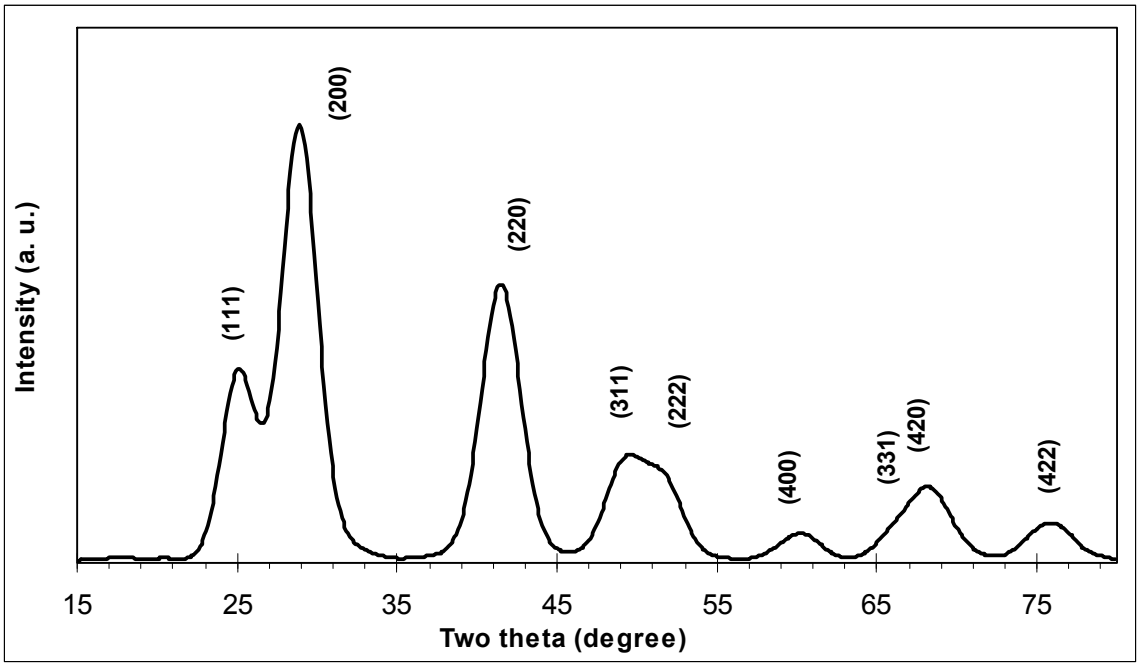


Figure S7A. The XRD pattern of our small-sized PbSe NCs. The PbSe NCs were prepared from a batch (as shown in Figure S8B left) with the feed molar ratio of 1.47DPP-to-1Pb and 1Pb-to-2.5TBPSe, and [Pb] of ~ 71 mmol/kg. The growth temperature was 50 °C and the growth period was 25 min. The synthetic condition was identical to that shown in Figure 2 Batch c. The high synthetic reproducibility is worthy of notice. The PbSe NCs exhibited their first excitonic absorption peaking at 1081 nm. The XRD pattern obtained matches perfectly that of the cubic rock salt crystal structure obtained from bulk PbSe.

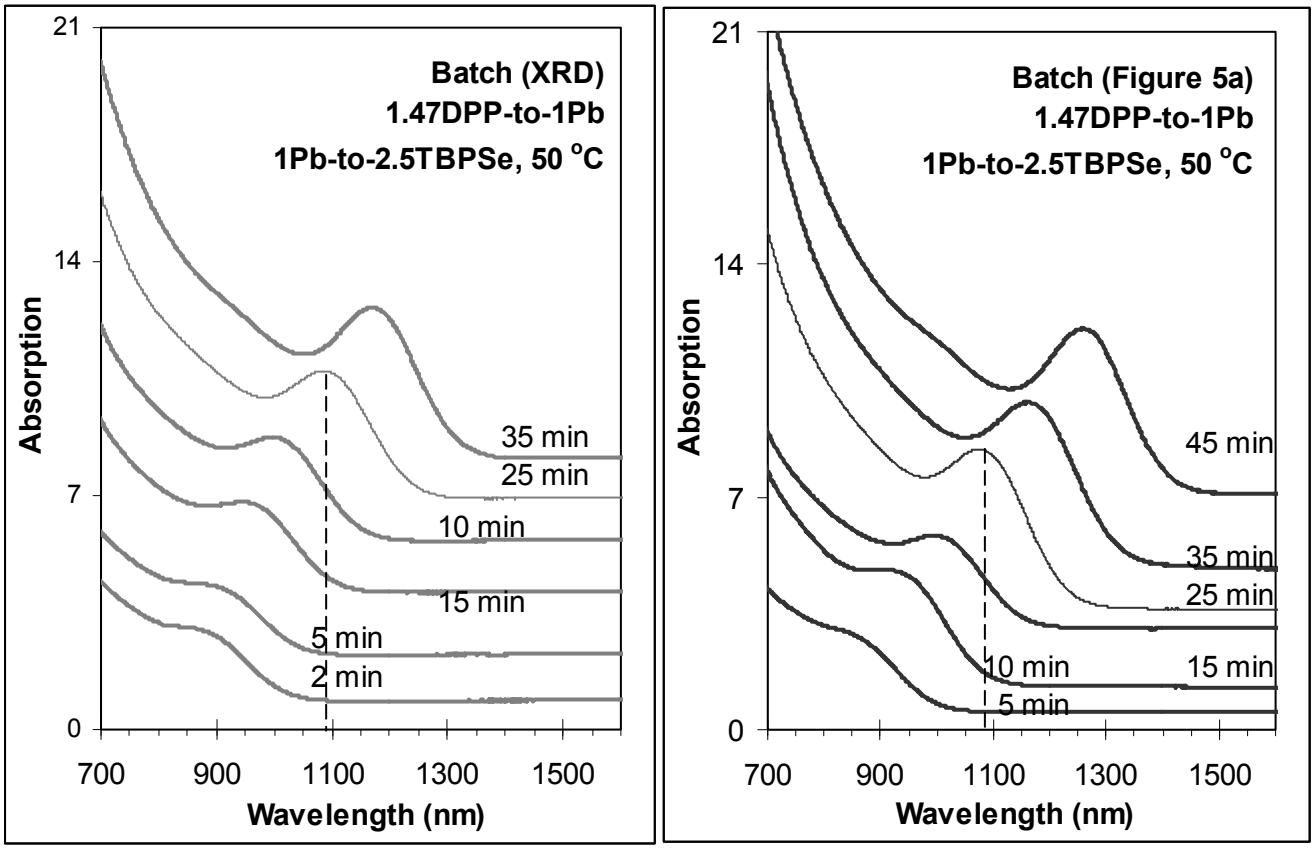


Figure S7B. The high synthetic reproducibility of our non-injection-based approach is worthy of notice. Here (an example as Method (5)), Batch left and Batch right had the identical synthetic condition. The 25-min PbSe NCs from Batch left was used for XRD shown in Figure S7A. Temporal evolution of the absorption (offset) of the PbSe NCs from the two batches. The absorption spectra were normalized to 1.0 gram of crude growth mixtures and dispersed in 1.0 mL TCE.

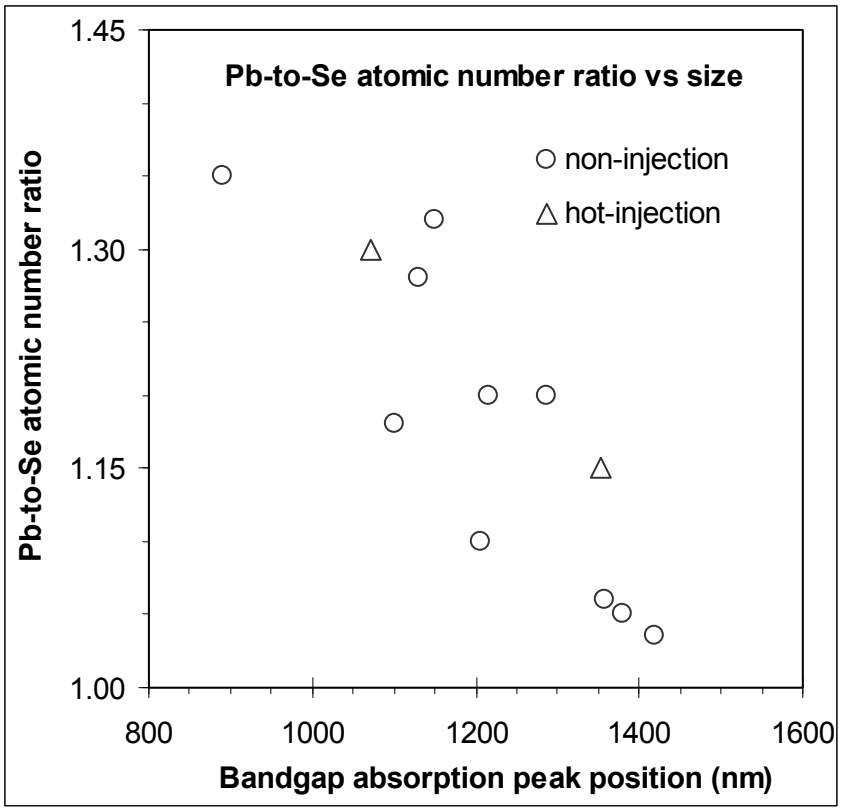


Figure S7C. The Pb-to-Se atomic number ratio of the PbSe NCs prepared. The data were from EDX.