# Influence of Arylalkyl Amines on the Formation of Hybrids CsPbBr<sub>3</sub> Nanocrystals via Modified LARP Method.

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## I. Synthesis and characterization Methods

#### A. <u>Materials</u>.

Lead(II) bromide (PbBr<sub>2</sub>, 99.99%), cesium carbonate (Cs<sub>2</sub>CO<sub>3</sub>, 99.99%), oleic acid (OA, 90%), phenylethylamine (PEA, C<sub>8</sub>H<sub>11</sub>N,  $\leq$ 100 %), dimethylformamide (DMF, 99.8% anhydrous), toluene ( $\geq$ 99.9%), tetra-octylammonium bromide (TOABr, 98%) were sourced from Sigma-Aldrich.

#### B. Synthesis.

Colloidal perovskite CsPbBr<sub>3</sub> NCs were fabricated by an adapted LARP technique but instead of using a pure toluene solution to fabricate NCs.

- The method to get highly calibrated CsPbBr<sub>3</sub> NCs consists of three main steps :
  - In the first one, a toluene solution of CsPbBr<sub>3</sub> NCs, so-called **PVK-TOA**, is prepared.
  - In parallel, a pre-SB solution is synthesized, containing PbBr<sub>2</sub>, OA, Cs<sub>2</sub>CO<sub>3</sub>, and AAA.
  - Then, a small volume of pre-SB solutions (2,5 % in volume of PVK-TOA) is added to PVK-TOA at RT to form the final monodisperse perovskite-NCs (AAA-NCs solutions).

**CsPbBr<sub>3</sub> NCs precursors stabilized by TOABr without AAA (PVK-TOA solution):** NCs were prepared by following the procedure reported by Wei *et al.*<sup>1</sup> PbBr<sub>2</sub> (0.1 mmol) and TOABr (0.1 mmol) were mixed in toluene (10 mL) and stirred until the full dissolution of reagents by slightly heating (45°C). When the PbBr<sub>2</sub> is totally dissolved, the solution is cooled at 0°C. – **vial A**. In parallel, Cs<sub>2</sub>CO<sub>3</sub> (2.5.10<sup>-5</sup> mol) was dissolved in OA (1 mL) – **vial B**. Then **vial B** was poured out **vial A** in one shot at room temperature and stirred for 5 min. Then NCs precursors were recovered after centrifugation at 10000 rpm for 20 min, at 5°C, and redispersed in toluene (10 mL).

#### Pre-AAA solutions in DMF vial :

General Pre-AAA solution: PbBr<sub>2</sub> (1.10<sup>-4</sup> mol) and OA (0.122 mL) were solubilized in DMF (1mL) at 35°C – vial C. Cs<sub>2</sub>CO<sub>3</sub> (2.5.10<sup>-5</sup> mol) and OA (0.122 mL) were solubilized in DMF (1mL) – vial D. Vial C and D were mixed together at room temperature. Then, AAA (molarity used for most AAAs, with the corresponding ratio of AAA/Pb<sup>2+</sup> (from SA) is reported in table below) was added to the mixture and the solution was stirred for 5 min to homogenize the solution.

Final CsPbBr<sub>3</sub> NCs (AAA-NCs solutions at different ratios) were used without other treatment:

• **Pre-AAA** solutions (0.125 mL) were poured onto **PVK-TOA** (5 mL) and stirred for a minimum of 10 min to complete the reaction and to get the final **AAA-NCs solutions**.

<sup>&</sup>lt;sup>1</sup>Homogeneous Synthesis and Electroluminescence Device of Highly Luminescent CsPbBr<sub>3</sub> Perovskite Nanocrystals. S. Wei, Y. Yang, X. Kang, L. Wang, L. Huang, D. Pan, *Inorg. Chem.* **2017**, *56*, 2596–2601; DOI: 10.1021/acs.inorgchem.6b02763

Acronyms of Samples	Name of AAA	Amount of AAA	mol of AAA	Ratio AAA/Pb2+
PEA NCs	Phenylethylamine	50 μL	4,78E-05	1,0
PPA NCs	3-Phenylpropylamine	56 μL	4,73E-05	0,9
PMA NCs	Benzylamine	43 μL	4,72E-05	0,9
PPMA NCs	4-Phenylbenzylamine	36 mg	2,36E-05	0,5
Naph-PMA NCs	1-Naphthylmethylamine	29 μL	2,38E-05	0,5
tBu-PMA NCs	4-tert-Butylbenzylamine	68 μL	4,65E-05	0,9
CF <sub>3</sub> -PMA NCs	4-(trifluoromethyl)benzylamine	55 μL	4,67E-05	0,9
MeO-PMA NCs	4-Metoxybenzylamine	30 μL	2,78E-05	0,6
Thio-MA NCs	2-(Aminomethyl)thiophene	39 μL	4,51E-05	0,9
Thio-EA NCs	2-(2-Aminoethyl)thiophene	46 μL	4,95E-05	1,0
MeO-PEA NCs	2-(4-Methoxyphenyl)ethylamine	30 μL	2,45E-05	0,5
CF <sub>3</sub> -PEA NCs	2-(4-Trifluoromethylphenyl)ethylamine	63 μL	4,00E-05	0,8
DPEA NCs	2,2-Diphenylethylamine	60 mg	3,65E-05	0,7
MPMA NCs	1-Phenylethylamine	51 μL	4,75E-05	0,9
TPMA NCs	Triphenylmethylamine	51 mg	2,36E-05	0,5
DPPA NCs	3,3-Diphenylpropylamine	63 mg	3,58E-05	0,7
MPEA NCs	2-Methyl-2-phenethylamine	22 μL	1,85E-05	0,4
DPMA NCs	Benzhydrylamine	69 μL	4,80E-05	1,0
AmAc1 NCs	Homophenylalanine	36 mg	2,41E-05	0,5
AmAc2 NCs	Baclofen	42,5 mg	2,39E-05	0,5

Table SI-I – Acronyms of Samples, Names of AAA and amount (in mL or mg) used in pre-AAA solutions

#### C. <u>Characterizations.</u>

NCs were observed and analyzed by transmission electron microscopy with a Microscope JEM 2100 Plus (JEOL) and an accelerating voltage of 200 kV. High-resolution TEM (HRTEM), high-angle annular dark field scanning TEM (HAADF-STEM) images, and energy-dispersive X-ray (EDX) spectroscopy were taken with a Microscope Titan3 G2 80-300 (FEI ThermoFisher) operating at 100 kV. The XRD experiments were performed on a Malvern Panalytical Aeris Research Edition, with a sample changer of 6 positions and a PIXcel 1D detector. The working power is 300 W (30 kV - 10 mA). The used radiation is copper Kalpha1+2 (approx. 1.5406 angstroem). Samples were placed on a Si (510) sample holder in order to reduce background. Data were measured from 2 to 60° in 2Theta with a total scan time of 10 min per scan with a rotation of the sample during acquisition. The UV–vis absorption spectra of NCs were recorded using a UV–vis spectrometer (Perkin-Elmer LAMBDA 950 UV/Vis/NIR Spectrophotometer) over the wavelength from 300 to 700 nm.

Emission and decay time-based measurements were registered by Edinburgh Instruments FS5 spectrofluorometer on the principle of the steady-state mode and the time-correlated single-photon counting (TCSPC) mode, respectively. The lifetime decay was measured with the module EDEDEPL-405 405 nm ((±5 nm) picosecond pulsed diode laserTypical pulse width: 60 ps @ 10 MHz Rep rate: 20 kHz - 20 MHz) coupled with FS5 spectrofluorometer. The internal quantum efficiencies of NCs were quantitatively determined using an integrating sphere-equipped spectrofluorometer (FS5, Edingburg). Fluorescence excitation and emission spectra, and quantum yield values were obtained using an Edinburgh Instrument FS5 fluorescence spectrometer. The spectra were recorded in right angle mode using quartz cuvettes with a 1 cm path length. The fluorescence quantum yield values were obtained using the integrating sphere sample cassette available for the FS5 spectrometer. The Fluoracle software was used to calculate the absolute quantum yield values by comparing the blank and the sample spectra.

Samples	τ1	1%	τ2	2%	τ3	3%	xchi2	QY	τampl	Kr	Knr	Kr/I	Knr
TBU-PIVIA PPA	1,85 QY 2,4	13,3 15, <b>4</b> 1	8,4 8,6	46,3 <b>72</b> 55,3	35,6 2% 32	40,4 <b>53</b> ,3	<sup>1,16</sup> 3% 1,1	xchi2 <sub>80%</sub> τ	ver 18,52	0,044 Kr 0.05	0,01	Knr	4,400
RNEK TARA	11%	13 <b>,17,9</b>	<b>g,6</b> ,5	48912	<b>5</b> 5,8	3660	19,71,12	1,15 78%	25,84 18.15	0,004 0.043	0,034 0.012	0,124	3,583
Bea	29,05%	14 <b>2</b>	8 <del>1</del> ,£67	5 <b>7,85</b>	54,95	3345	28,91,16	1,11 <sub>71%</sub>	24,16 17.4	0,025 0.041	0,016 0.017	1,564	2.412
RPA-PEA	80%	14 <b>2,4</b>	8 <sup>1</sup> 634	49 <b>856</b>	55 <b>34</b> 25	<b>32</b> .2	29,3 <sub>1.14</sub>	1,1 <sub>70%</sub>	23,58 16.55	0,034 0.042	0,008 0.018	4,000	2.333
PMAEA	2:5%	1,85	81368	57 <b>8<sub>8</sub>3</b>	4 <b>6</b> 85	34,4	37,71.1	1,14 <sub>70%</sub>	27,83 12.94	0,023 0.054	0,013 0.02	1,778	2,348
BRIMA	59%	131,5	18,7	7,1	34.9	33,4	32,21.14	1,12 <sub>64%</sub>	26,48 17.25	0,022	0,015	1,439	1 762
Naph-PMA	62%	2,05	8 <sup>14</sup>	_8,16	51 A	37,35	34,61,13	1,16 64%	29,73 16.91	0,021 0,038	0,013 0,021	1,632	1 810
tBu-PMA	81%	1,85	13,3	58,4	34.33	35,6	40,41,16	1.16 62%	29,44	0,028	0,006	4,263	1 636
CF <sub>a</sub> -PMA	56%	16, <b>41</b>	7.85	54,53	41,3 32:4	39,4	43 1.11	1,17 <sub>61%</sub>	34,36	0,016 0,030	0,013 0.028	1,273	1 571
MeQ_PMA	78%	12,14	13,7	<u>\$</u> ,7	48,1	35,8	38,2112	1,12 59%	29,01 14,53	0,027 0.041	0,008 0.028	3,545	1 464
ThigMA	A2%	51,6	22,11	_ <b>6,</b> 55	51,52	33,8	26,37 17	1,15 56%	25,62 22.3	0,016 0.025	0,023 0.02	0,724	1,250
Thio-EA	55%	,2,13	18,2	7,9	47,8	38,7	34 1 11	1,11	31,17	0,018	0,014 0.020	1,222	1 221
MeO-PEA	70%	1,8	14,23	8,03	49,5	34	36,2	1,14	27,26 22,26	0,026	0,011 0,020	2,333	1,231
ÇE3-PEA	70%	2,52	15,04	8,56	57,8	28	27,2,15	1,1 42%	19,82	0,035 0,02	0,015	2,333	0,800
DREA	45%	19,25	9,33	42,9	238	47,9,	1,14, 12	1,8 41%	32,77	0,055	-0,024 0,040	-2,250	0,717
MPMA	41%	2.02	15,3	<b>8</b>	50	38,1	34,7	1,12	30,60 34.80	0,013 0,016	0,019 0,03	0,695	0,070
ТРМА	5%	ud	75	1 u d	45	ų į	1.08	10%	24,85	0,010	0,02		0,007
DPPA	71%	2,05	_14	8,16	51,4	37,35	34,6,115	1,16	29,73	0,024 0,005	0,010	2,448	0,230
MPEA	64%	20,5	14,2	8,5	51,8	35,9	34 34	1,13	28,17	0,023	0,013	1,778	0,125
DPMA	8%	ud		ud		ud		878					
<b>APA</b>	19%	19,2	<i>u</i> g <sub>5</sub>	80,84	ua		1,08	1.08 5,00%	44,96	0,024	-0,002	13,500	<u> </u>
Magenne	<b>40</b> %	<sup>14</sup> 26,6	<sup>8</sup> 2,9	48 <b>13</b>	358	387,4	52,1 <sup>1,1</sup>	1,21 <sup>56%</sup>	30,8 <mark>8,1</mark>	0,0 <b>9,9</b> 84	0,0926	0,667	B4
ecart-type	0,32	5,32	1,38	8,15	3,52	12,91	0,03	20%	5,90	0,014	0,016	1,2	.05
		QY < 20 %		40% <qy< 6<="" td=""><td>9%</td><td></td><td>70% <qy< 77%<="" td=""><td></td><td>QY&gt; 3</td><td>77%</td><td></td><td></td><td></td></qy<></td></qy<>	9%		70% <qy< 77%<="" td=""><td></td><td>QY&gt; 3</td><td>77%</td><td></td><td></td><td></td></qy<>		QY> 3	77%			

 Table SI-2
 Optical data for AAA-NCs

The average lifetime  $(\tau_{ave})$  can be calculated using the following equation :  $< \tau(aver) > = \frac{\sum \tau(i)\alpha(i)}{\sum \alpha(i)}$ 

 $K_r = (PLQY) / <\tau_{aver} >; K_{nr} = (1-PLQY) / <\tau_{aver} >$ 

 ${}^{s}\!\alpha_{i} \text{ represents the amplitude of the i-th lifetime component.} \\ {}^{s}\!<\!\tau_{amp}\!> = \Sigma\tau_{i}\alpha_{\prime}/\Sigma\alpha_{\nu} \ k_{r} = \mathsf{PLQY}/<\!\tau_{amp}\!> \text{ and } k_{rr} = (1-\mathsf{PLQY})/<\!\tau_{amp}\!>.$ 

#### II. XRD analysis of AAA-NCs

XRD analysis has been used to characterize the structures of NCs perovskites. The different samples are prepared by drop-casting the NCs solution on a glass plate and then drying them at room temperature, contrary to TEM observation, where the solution is dropped onto the copper grid and NCs are deposited onto the grid by suction of solvent with a filter paper. This point will be crucial to understand the XRD results.



**Scheme SI-1** – (a) DRX of PVK-TOA, (b) DRX Data for Structure monoclinic (5.827 x 5.891 x 5.827, beta=89.65) of CsPbBr<sub>3</sub>, Reference ICSD 18-364.

#### III. Histogram sizes of AAA-NCs

<u>NCs sizes and distance separations estimate.</u> NCs size was estimated from TEM pictures using the Julia programming language<sup>2</sup>, the *ImageSegmentation.jl* library and a watershed algorithm<sup>3</sup>, like described in our previous communication.<sup>4</sup>

**Table SI-3** – Average sizes and standard deviation ( $\sigma$  size) in nm of AAA-NCs by TEM analysis

Samples	Average size	σ size		
PVK TOA	38	17,1		
PMA	11,83	3,83		
MeO-PMA	9,91	3,13		
TBu-PMA	12,4	5,26		
CF <sub>3</sub> -PMA	9,4	3,25		
Thio-MA	13,2	3,54		
4-PPMA	9,74	2,52		
Naph-PMA	11	3,55		
PEA	10,1	1,23		
CF <sub>3</sub> -PEA	13,7	2,86		
MeO-PEA	9,87	3,76		
Thio-EA	nd	nd		
PPA	8,14	4,15		
МРМА	14,02	2,97		
MPEA	11,04	3,84		
DPEA	17,4	8,93		
DPPA	11,9	3,5		
ТРМА	nd	nd		
DPMA	nd	nd		
AA2	11,7	1,5		
AA1	nd	nd		

<sup>2</sup> J. Bezanson, A. Edelman, S. Karpinski and V. B. Shah, SIAM Review, 2017, 59, 65–98.

<sup>3</sup> R. C. Gonzalez and R. E. Woods, in *Digital Image Processing*, Prentice Hall, 2nd edn., 2002, pp. 567–642.
4 . C.R. Mayer, H. Levy-Falk, M. Rémond, G. Trippé-Allard, F. Fossard, M. Vallet, M. Lepeltier, N. Guiblin, J.-S. Lauret, D. Garrot, E. Deleporte, *Chem. Commun.*, 2022, *58*, 5960-5963

# IV. Optical spectroscopies of AAA-NCs











Figure SI-2 – UV-Vis spectra of -a- AmAc1 and AmAc2, and -b- DPMA, TPMA, MPMA and PVK -NCs.

#### B. Photoluminescence studies of AAA-NCs



Figure SI-3 – PL Spectra of core PMA-NCs series.



Figure SI-4– PL Spectra of calibrated NCs, zoom view for PL of TPMA and DPMA NCs.



Figure SI-5 – PL Spectra of others calibrated of core PEA-NCs.

#### c. Time resolved photoluminescence studies of AAA-NCs



Figure SI-6 – TRPL trace of NCs for series – a- PMA cores, – b- others phenylakylamines, and -c-AmAc1

# V. Library of AAA-CsPbBr<sub>3</sub> NCs synthesized with their characterizations

### 1- PVK TOA NCs

b- $\lambda_{max}$  = 517 nm FWHM = 18 nm Size = 38.0 ± 17.1 nm QY = 11 % 



C-

a-



**Figure SI-7** -a- General data, -b- XRD of PVK-TOA and PEA NCs; -c – Typical TEM (200 kV) pictures of PVK TOA NCS with scale bar 200 nm

#### 2- PMA-NCs - Benzylamine NCs



**Figure SI-8** – In left top, General data, in right top, XRD pattern and in botton, TEM (200 kV) pictures of **PMA-NCS** with scale bar – (a) 200 nm – (b) 200 nm – (c) 50 nm and – (d) 100 nm.



Figure SI-9 – (a) Histogram of NCs sizes and Gaussian fit and (b) Identified NCs areas of PMA-NCs.

#### 3- MeOPMA-NCs - 4-Methoxybenzylamine NCs



Figure SI-10 – In left top, General data, in right top, XRD pattern and in botton, TEM (200 kV) pictures of MeO-PMA NCS with scale bar – (a) 200 nm and – (b) 200 nm. b-







#### 4- tBuPMA-NCs - 4-tert-Butylbenzylamine NCs



**Figure SI-12–** In left top, General data, in right top, XRD pattern and in botton, TEM (200 kV) pictures of tBuPMA-NCS with scale bar – (a) 100 nm – (b) 20 nm – (c) 100 nm and – (d) 200 nm.





**Figure SI-13** – (a) Histogram of NCs sizes and Gaussian fit and (b) Identified NCs areas of tBuPMA-NCs.

#### 5- CF<sub>3</sub>PMA-NCs - 4-(Trifluoromethyl)benzylamine NCs



**Figure SI-14** In left top, General data, in right top, XRD pattern and in botton, - TEM (200 kV) pictures of CF<sub>3</sub>PMA-NCS with scale bar – (a) 100 nm – (b) 200 nm – (c) 200 nm and – (d) 200 nm.

b-





Figure SI-15 – (a) Histogram of NCs sizes and Gaussian fit and (b) Identified NCs areas of CF₃PMA-NCs.

### 6- ThioPMA-NCs - 2-(Aminomethyl)thiophene NCs



**Figure SI-16** In left top, General data, in right top, XRD pattern and in botton, – TEM (200 kV) pictures of ThioMA NCS with scale bar (a) 200 nm and – (b) 100 nm.

#### 7- PPMA-NCs - 4-Phenylbenzylamine NCs



**Figure SI-17-** In left top, General data, in right top, XRD pattern and in botton, -TEM (200 kV) pictures of PPMA-NCS with scale bar – (a) 100 nm – (b) 100 nm – (c) 50 nm and – (d) 500 nm.



**Figure SI-18** – (a) Histogram of NCs sizes and Gaussian fit and (b) Identified NCs areas of PPMA - NCs.

#### 8- NaphPMA-NCs - 1-Naphtylenemethylamine NCs



**Figure SI-19** In left top, General data, in right top, XRD pattern and in botton, – TEM (200 kV) pictures of NaphPMA-NCS with scale bar – (a) 200 nm – (b) 200 nm – (c) 100 nm and – (d) 100 nm.

b-





**Figure SI-20** – (a) Histogram of NCs sizes and Gaussian fit and (b) Identified NCs areas of NaphPMA-NCs.

#### 9- CF<sub>3</sub>PEA-NCs - 2-(4-Trifluoromethylphenyl)ethylamine NCs



**Figure SI-20-** In left top, General data, in right top, XRD pattern and in botton, – TEM (200 kV) pictures of CF<sub>3</sub>PEA-NCS with scale bar – (a) 200 nm and – (b) 100 nm.

b-





**Figure SI-21** – (a) Histogram of NCs sizes and Gaussian fit and (b) Identified NCs areas of  $CF_3PEA-NCs$ .



**Figure SI-22-** In left top, General data, in right top, XRD pattern and in botton, – TEM (200 kV) pictures of MeOPEA-NCS with scale bar – (a) 200 nm and – (b) 100 nm.

b-





Figure SI-23 – (a) Histogram of NCs sizes and Gaussian fit and (b) Identified NCs areas of MeOPEA-NCs.

#### 11- **ThioEA-**NCs - 2-(Aminoethyl)thiophene NCs



**Figure SI-24** - In left top, General data, in right top, XRD pattern and in botton, – TEM (200 kV) pictures of ThioEA-NCS with scale bar – (a) 500 nm and – (b) 200 nm.



**Figure SI-25** - In left top, General data, in right top, XRD pattern and in botton, – TEM (200 kV) pictures of PPA-NCS with scale bar – (a) 100 nm and – (b) 200 nm.



Figure SI-26 – (a) Histogram of NCs sizes and Gaussian fit and (b) Identified NCs areas of PPA-NCs.



**Figure SI-27** - In left top, General data, in right top, XRD pattern and in botton, – TEM (200 kV) pictures of MPMA-NCS with scale bar – (a) 500 nm and – (b) 200 nm.

#### 14- **MPEA-**NCs - N-methyl-2-phenylpropan-1-amine NCs



**Figure SI-28** – In left top, General data, in right top, XRD pattern and in botton, - TEM images of solution of MPEA-NCs recorded on -a- JEOL (200 kV) with a scale bar of 200 nm and -b- HR BF-STEM picture with a scale bar 10 nm.



**Figure SI-29** – (a) Histogram of NCs sizes and Gaussian fit and (b) Identified NCs areas of MPEA - NCs.



**Figure 30** - a) XRD DPEA-NCs crude solution (SB) before centrifugation b) DPEA-NCs precipitate after centrifugation of the crude solution and c) DPEA-NCs filtrate after centrifugation of the crude solution



**Figure SI-31** – TEM (200 kV) pictures of DPEA-NCS with scale bar 200 nm with a TEM (200 kV) (a) and (b), and – (c),(d) by HR HAADF-STEM scale (c) 20 nm and (d) 10 nm.



Figure SI-32 – EDX elemental mappings for DPEA-NCs



Figure SI-33 – Histograms of NCs sizes and Gaussian fit (a) and (c), Identified NCs areas (b) and (d) of DPEA-NCs in for both kinds of NCs populations.



**Figure SI-34** – In left top, General data, in right top, XRD pattern and in botton, TEM (200 kV) pictures of DPPA-NCS with scale bar – (a) 200 nm and – (b) 500 nm.



Figure SI-35 – (a) Histogram of NCs sizes and Gaussian fit and (b) Identified NCs areas of DPPA-NCs.



**Figure SI-36** – In left top, General data, in right top, XRD pattern and in botton, TEM (200 kV) pictures with scale bar 200 nm of DPMA-NCs



 $\label{eq:lambda} \begin{array}{l} \lambda_{max} = 522 \ nm \\ FWHM > 19 \ nm \\ \mbox{Size} = undetermined \\ QY = undetermined \end{array}$ 





**Figure SI-37** – In left top, General data, in right top, XRD pattern and in botton, TEM (200 kV) pictures with scale bar 200 nm of TPMA-NCs

#### 19- **AmAc1-**NCs - Homophenylalanine NCs





**Figure SI-38** – In left top, General data, in right top, XRD pattern and in botton, TEM (200 kV) pictures of AcAm1-NCs (scale bar 200 nm).

#### 20- AmAc2-NCs - Baclofen NCs



 $\lambda_{max} = 519 \text{ nm}$ FWHM = 19 nm Size = 11.7 ± 1.5 nm QY = 40 %







**Figure SI-39** – In left top, General data, in right top, XRD pattern and in botton, HAADF-STEM of AcAm2-NCs, scale bar 100 nm and 20 nm.



Figure SI-40 – EDX elemental mappings for AmAc2-NCs

# VI- Influence of electronic beam on Pb° Nanoparticles onto the nanocrystal surfaces.



**Figure SI-41** – HAADF-STEM pictures of PEA-NCs observed at different exposures times (scale bar 20 nm) -a- at t= 0 min; -b- t= 1 min; and -c- at t= 2 min.

In the Fig. SI 41, the three images clearly show the disappearance of lead nanoparticles on the surface of nanocrystals under the electron beam as the observation time increases.

# VII- Highlighting of PbO structures



**Figure SI-42-** HAADF-STEM pictures in high resolution of NCs showing PbO ribbons and superposition of schematic representation of PbO -a- from DPEA-NCs with a scale bar 5 nm showing, -b- with a zoom on a selected area – c – from PEA-NCs and – d- with an increase zoom of the selected zone, on the picture c.



**Figure SI-43** – HAADF-STEM pictures in high resolution of NCs showing PbO ribbons and superposition of schematic representation of PbO from PEA-NCs (with ratio PEA/Pb<sup>2+</sup> = 4) -a- scale bar 10 nm – b- zoom view of the area, -c- other view and – d- schematic view of PbO ribbons.