## **Supporting information for**

## In-situ high-energy synchrotron X-ray diffraction revealing precipitation reaction kinetics of silver ions with mixed halide ions

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Fig. S1 GSAS calibration results for the  $LaB_6$  standard powders in the reactor. The standard XRD pattern of  $laB_6$  has been given for comparison, while the difference between the observed data and calculated data is only 1.27%.



Fig. S2 The contribution of the instrument to peak broadening ( $\delta i$ ) as a function of 2-theta angle, which were calculated based on the LaB<sub>6</sub> standard.

Description of the method to calculate the particle size of the nanocrystals: The LaB<sub>6</sub> standard has been measured and fitted by GSAS software (Fig. S1), as a result, the parameters of (U, V and W) of the profile shape function can be obtained; and the contribution of the instrument to peak broadening ( $\delta_i$ ) can be calibrated via the following equation:

$$\delta i = U tan^2 \theta + V tan \theta + W \tag{1}$$

And the contribution of the instrument to peak broadening ( $\delta_i$ ) as a function of 2–theta angle has been shown in Fig. S2. The FWHM value ( $\delta m$ ) extracted from the XRD patterns recorded from the synthesis reaction was corrected from considering the instrumental contribution. The corrected FWHM value ( $\delta_s$ ) can be calculated via the following function:

$$\delta s = \sqrt{\delta m^2 - \delta i^2} \tag{2}$$

With the use of corrected FWHM, the lateral dimensions of the nanocrystals were calculated by employing the Scherrer equation:

$$L = \frac{K\lambda}{(\delta s)cos\theta} \tag{3}$$

Where *L* is the mean size of the lateral dimensions of the nanocrystals; *K* is the dimensionless shape factor, with a value close to 0.9 (the peaks were fitted by Gaussian function);  $\lambda$  is the X-ray wavelength;  $\delta s$  is the corrected FWHM value after subtracting the instrumental contribution, in radius.  $\theta$  is the Bragg angle.

An example to calculate the lateral dimensions of the crystalline domains (particle size) in the  $AgCl_{0.44}Br_{0.56}$  nanoparticles along the (200): For the Synchrotron X-ray diffraction pattern taken *in-situ* at 100 s during precipitation reaction process with an injection rate (AgNO<sub>3</sub>) of 1ml·min<sup>-1</sup>, ( $\lambda = 0.1771$  Å), the measured FWHM was 0.0108, Then the contribution of the instrument to peak broadening ( $\delta i$ ) at 2 $\theta$ =3.58° was 0.0080 (Fig. S2). Then the measured FWHM was calculated to be 0.0073 (1.273E-4 in radius). Consequently, according to the Sherrer equation, the mean size of the lateral dimensions of the nanocrystals along (200) plane was calculated to be 125.16 nm.



Fig. S3 XRD patterns taken right after the complete addition of  $AgNO_3$  to the reaction solution as the reaction shown in Fig. 2. In this reaction, the injection rate of  $AgNO_3$  solution was 1 mL·min<sup>-1</sup>. The wavelength of the X-ray was 0.1771Å.



**Fig. S4** Conversion to  $AgCl_xBr_{1-x}$  (, % in mass, data extracted from the peak area of the (200) peak in Fig. 2).



**Fig. S5** XRD pattern recorded from the reaction shown in Fig. 5 at 300 s. The Gaussian fitting highlights the strategy for extracting quantitative information of two individual  $AgCl_xBr_{1-x}$  phases in the product mixtures.

**Table. S1** Kinetic parameters for the reaction the reaction solution containing KBr and NaCl (with the molar ratio ( $[Cl^-]/[Br^-])=1:1$ ) and AgNO<sub>3</sub> that was added at an injection rate of 1 mL·min<sup>-1</sup>.

<b>Equation</b> $y = A_2 + \frac{(A_1 - A_2)}{(1 + \exp(\frac{x - x_0}{dx}))}$				
A <sub>1</sub>		$\mathbf{A}_{2}$	<i>x</i> <sub>0</sub>	dx
0		3.81	72.4	5.64

\*y and x correspond to the axes of peak area and reaction time respectively. A<sub>1</sub> represents the amount of peak area at t=0 s; A<sub>2</sub> represents the amount of peak area at t=96 s;  $x_0$  indicates the time when half of AgCl<sub>0.44</sub>Br<sub>0.56</sub> nanoparticles has been produced.